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EVALUATION OF HAZARDOUS WASTE INCINERATION IN AN AGGREGATE KILN: FLORIDA SOLITE CORPORATION

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Consistent achievement of greater than 99.99% DRE was demonstrated for each POHC. Emissions of other pollutants ranged as follows: particulates - 4.4 to 6.5 kg/hr; HCl - 0.008 to 0.034 kg/hr; SO_2 - 72.2 to 99.6 kg/hr; NO_x - 1.9 to 11.7 kg/hr. Between 60 and 90% of the element chlorine is fed to the kiln from the waste fuel and scrubber influent water, while 95% of the chlorine is discharged from the process as chloride ion in the scrubber effluent water.

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FOREWORD

Today's rapidly developing and changing technologies and industrial products and practices frequently carry with them the increased generation of solid and hazarduos wastes. These materials, if improperly dealth with, can threaten both public health and the environment. Abandoned waste sites and accidental releases of toxic and hazardous substances to the environment also have important environmental and public health implications. The Hazardous Waste Engineering Research Laboratory assists in providing an authoritative and defensible engineering basis for assessing and solving these problems. Its products support the policies, programs and regulations of the Environmental Protection Agency, the permitting and other responsibilities of State and local governments and the needs of both large and small businesses in handling their wastes responsibly and economically.

This report describes a demonstration program on hazardous waste incineration in a wet process Portland Cement Kiln. It will be used to support technically defensible regulations in the evaluation of the future effort to develop the technology. The regulatory agencies as well as cement plants contemplating the use of waste fuels will find the results presented in this report helpful. Further information on this subject may be obtained by contacting the Thermal Destruction Branch, Alternative Technologies Division, HWERL.

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ABSTRACT

Aggregate kiln incineration of chlorinated liquid organic waste was investigated in a one-week program at Florida Solite Company. POHCs (toluene, tetrachloroethylene, methyl ethyl ketone, and methyl isobutyl ketone) were monitored in waste and stack emissions. In addition, stack emissions were monitored for particulate matter, particulate trace metals, HCl, SO_2, and NO_x. Process samples were collected and analyzed for trace metals and chloride. The destruction and removal efficiency of POHCs and the fate of trace metals and chloride ion in the kiln process were determined.

Consistent achievement of greater than 99.99% DRE was demonstrated for each POHC. Emissions of other pollutants ranged as follows: particulates - 4.4 to 6.5 kg/hr; HCl - 0.008 to 0.034 kg/hr; SO_2 - 72.2 to 99.6 kg/hr; NO_x - 1.9 to 11.7 kg/hr. Between 60 and 90% of the element chlorine is fed to the kiln from the waste fuel and scrubber influent water, while 95% of the chlorine is discharged from the process as chloride ion in the scrubber effluent water.

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SECTION 1

INTRODUCTION

Cofiring of hazardous wastes in high temperature industrial processes is an attractive alternative to hazardous waste incineration. The alternative makes use of the waste's heat content. Many processes including cement and dolomite kilns, glass furnaces, steel furnaces, and some industrial boilers operate at temperatures and residence times similar to those required for incinerators dedicated to incineration of hazardous wastes and thus offer opportunities for cofiring. In addition to the savings derived from the heat value in the waste fired, the use of existing industrial equipment, provides an environmentally acceptable alternative to conventional hazardous waste disposal while not requiring the capital expenditure that would otherwise be required if a separate incinerator to process and incinerate a given amount of hazardous waste is to be built.

Aggregate kilns, because of their high enengy use, are an excellent example of this concept. Such kilns typically operate at temperatures over 1093°C (2000°F), have gas residence times in excess of 1.5 seconds, and have a highly turbulent combustion zone. However, the need still exists for data that shows the effect of hazardous waste cofiring on the emissions from the aggregate process.

Currently, the State of Florida Department of Environmental Regulation (DER), in implementing RCRA regulations, allows exemptions from hazardous waste incinerator emmission standards if the facility uses the waste as a fuel for the purpose of recovering useable energy. The Florida Solite Corporation located in Green Cove Springs, Florida, petitioned the Florida DER for a change in

their air pollution permit to allow the burning of organic wastes as supplemental kiln fuel, thereby recovering energy from these wastes and offsetting the use of pulverized coal. The State of Florida DER and EPA Region IV requires testing of stack gases to meet new permit requirements. The EPA Region IV funded the testing through the EPA Industrial Environmental Research Laboratory-Cincinnati, which in turn contracted Monsanto Research Corporation to conduct the sampling and analysis. Conducted during the burning of liquid organic hazardous waste as supplemental fuel in Florida Solite's aggregate kiln, the sampling and analytical program was designed to determine the stack gas particulate emissions, the destruction of the principal organic hazardous constituents (POHCs) in the kiln, and the fate of heavy metals in the waste. Program results were to allow the Florida DER to decide whether the Solite Corporation could burn the new waste fuel under their existing permit, and if the existing regulatory exemptions are environmentally sound. Also, the results would provide additional hazardous waste incineration data for the EPA Industrial Environmental Research Laboratory - Cincinnati.

Sampling and analysis was conducted at Florida Solite Corporation on February 20 through 25, 1983, during the burning of coal and liquid organic hazardous waste as fuel supply. The sampling and analysis program included evaluation of: (1) the effects of cofiring coal and waste fuel on the destruction and removal efficiency (DRE) of principal organic hazardous constituents (POHCs), (2) the concentrations of particulate matter, SO_2, NO_x, HCl, and metals in the stack effluent, and (3) the fate, concentration, and distribution of metals and chlorine in the process streams.

The following sections of this report describe the site and its facilities, and the results of the sampling and analysis program.

SECTION 2

DESCRIPTION OF PROCESS AND WASTE FUEL SYSTEM

The Florida Solite Company operates an aggregate kiln in Green Cove Springs, Florida, which is located approximately 20 miles south of Jacksonville, Florida. Annual production of the expanded lightweight inorganic material made at this facility and used as aggregate in a cement mix is approximately 5.45×10^7 kg (60,000 tons) per year.

2.1 PROCESS DESCRIPTION

Plant facilities consist of (1) quarries from which raw clay material is extracted, trucked to the storage area, and fed to the clay feed hopper as needed for aggregate production; (2) three rotary kilns (of which only kiln No. 5 was in operation) which convert the clay into lightweight aggregate; (3) aggregate product handling and storage areas; (4) liquid hazardous waste fuel storage areas and fuel pumping system; (5) a coal rail and hopper feed system; (6) a kiln burner system for coal and waste fuel; and (7) a kiln emissions handling system including a cyclone, fan, wet scrubber, knockout chamber, and stack. Figure 2-1 illustrates the kiln plant layout including major equipment and the waste storage area. Figure 2-2 shows kiln No. 5 from the burner end. The figure also shows aggregate product (bottom right) and the waste fuel storage tanks, pump house, and stack (left). Figure 2-3 provides a view of kiln No. 5 taken from the stack with aggregate product handling shown in the background.

The process involves feeding the clay into a rotary kiln and heating it to a temperature of 1,093°C to 1,150°C (2,000°F to 2,100°F).

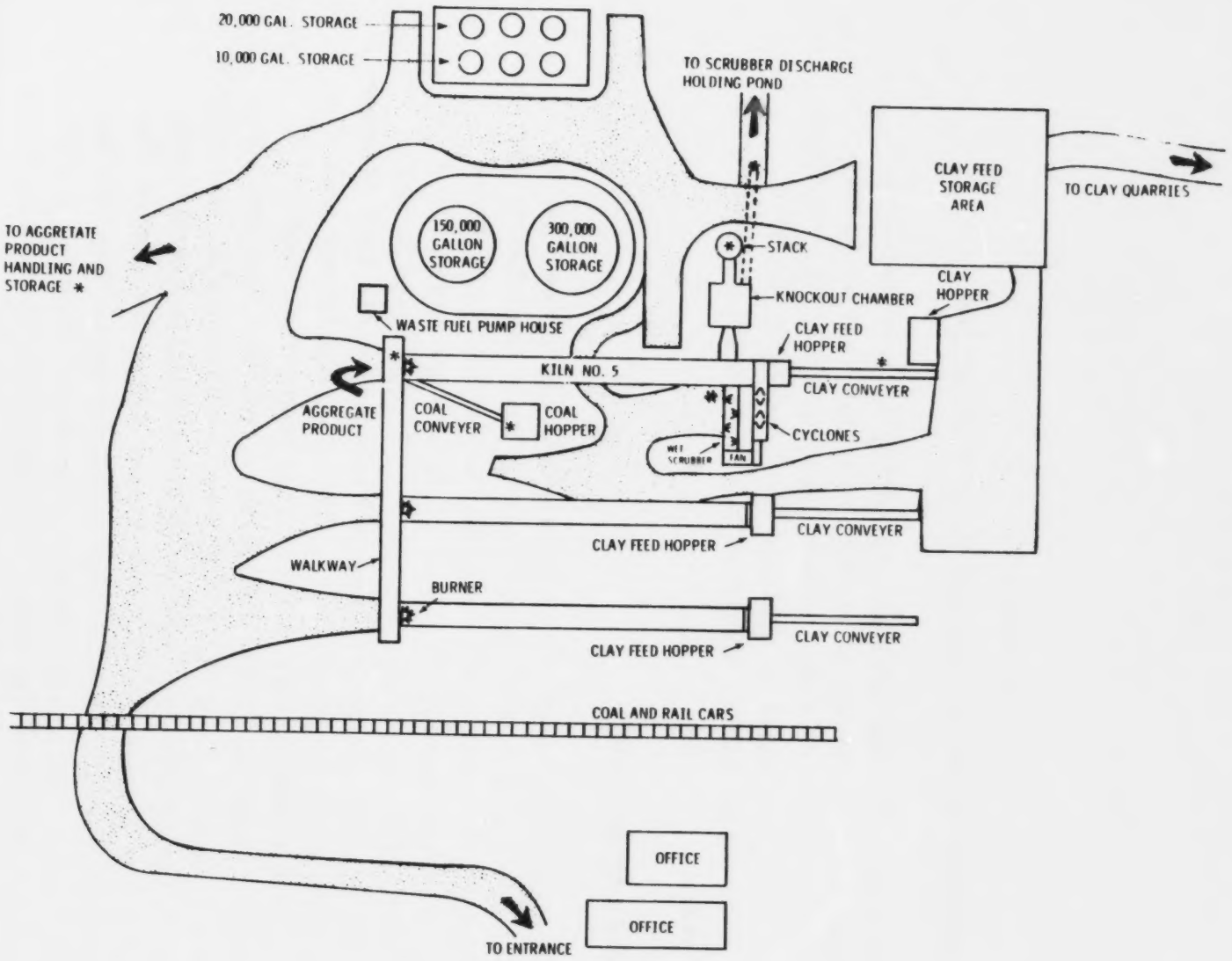

Figure 2-1. Florida Solite Site layout and sample locations (shown by asterisks).

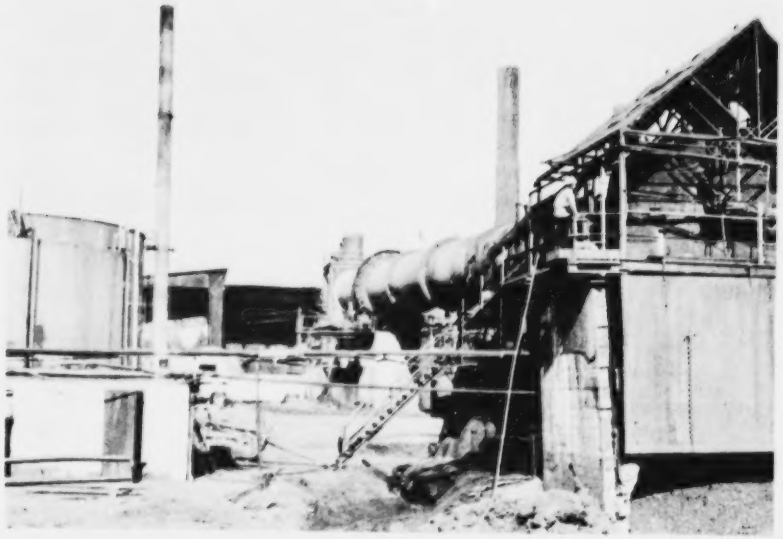

Figure 2-2. Kiln No. 5 (right), and waste fuel storage area and stack (left).

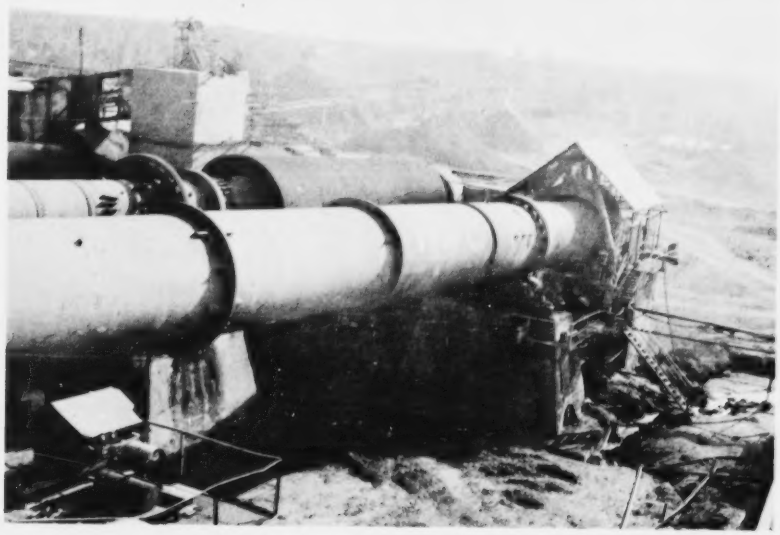

Figure 2-3. Kiln No. 5.

If the kiln temperature falls below that general range, the clay structure is not converted to the desired inorganic formation. If the temperature rises above the desired range, the material fuses, forming a heavy coating on the kiln interior that must be removed manually after stopping and cooling down the kiln. The only indication that proper temperature is being maintained in the kiln consists of visual observation that "solite" material is being produced and not some form of the other two undesirable alternatives.

The kiln, with refractory linings, is 2.7 m (9 ft) in diameter and 45.7 m (150 ft) long. The kiln rotates at 90 revolutions per hour, has a gentle slope (6.25 cm/m) to allow material to pass through by gravity. The kiln operates in a counter current flow pattern, i.e., solid materials travel in one direction and hot gases in the opposite direction. Clay is fed into the kiln at the upper end at a rate of approximately 12,260 kg/hr (27,000 lb/hr). At the opposite or lower end of the kiln, a mixture of coal and waste fuel is burned at rates of approximately 700 kg/hr (1,540 lb/hr) and 0.94 m^3/hr (248 gal/hr), respectively, to provide a heat input of approximately 13 MW (44 million Btu/hr). As the clay travels down the inclined rotating kiln, it passes through various temperature regimes which cause transformation of the clay into the lightweight aggregate product. The lightweight aggregate is produced at a rate of approximately 9,080 kg/hr (20,000 lb/hr). The energy required per ton of product is 4.4×10^6 Btu/ton. After heating and transformation in the kiln, the aggregate is graded and large clumps are crushed for sizing. The final product is stored in large piles until sold.

The kiln exhaust gases pass through a pair of cyclonic dust collectors, whose dust is recycled into the kiln, then into a horizontal cross-flow water scrubber where a series of water sprays removes the particulate matter and reduces the gas temperature from

about 370°C to 70°C (700°F to 160°F) before the gases reach the knockout chamber and are discharged through a fiberglass stack. Except for sampling of process streams, all sampling was conducted on emissions from the fiberglass stack. The scrubber water discharged from the knockout chamber is a mixture of raw steam and water with the entrapped particulate matter. This discharge stream is released to an open ditch which drains to a pond. There is no recycle of the scrubber water.

2.2 WASTE FUEL SYSTEM

The fuel used to fire the kiln is an unblended combination of crushed coal and waste organic liquids purchased from a sister corporation, Oldover. Florida Solite and Oldover Corporation are wholly owned subsidiaries of Solite Corporation, headquartered in Richmond, Virginia.

The liquid wastes, which are trucked directly from the generators have an approximate Btu value of 11,800 Btu/lb and consist primarily of solvents, alcohols, ethers, still bottoms, and a small fraction of chlorinated hydrocarbons. When the wastes are received at Florida Solite, each truckload is analyzed for halogen content, water content, flash point, and Btu rating. Any manifested wasteload that contains pesticides, PCBs, acids, caustics, cyanides, sulfides, mercaptans, electroplating wastes, or metal finishing wastes is rejected and returned to the generator. Accepted wastes are placed in 38- or 76-m^3 (10,000- or 20,000-gallon) storage tanks that are color-coded according to Btu value. The wastes are then blended to a fuel feed of consistent value and moved to a 300,000-gallon storage tank formerly used to store fuel oil. This large tank contains a 38-day supply of waste fuel. For the tests conducted by MRC, the waste fuel was blended to obtain a typical sample, stored in a 76 m^3 tank, and fed to the kiln from the 76 m^3 tank. The organic waste mixture makes up from 50% to

100% of the fuel used. During the test period, the waste fuel made up approximately 54% of the total fuel input. The remaining heat input was supplied by pulverized coal.

SECTION 3

SAMPLING AND ANALYSIS PROGRAM DESIGN AND TEST MATRIX

The sampling and analytical program was designed to identify the major pollutants from burning waste fuel in an aggregate kiln, quantify their respective emission rates, determine the destruction and removal efficiency (DRE) of the POHCs, and provide information for a mass balance around the process for metals and chlorine. Measured stack pollutants include POHCs (toluene, tetrachloroethylene, methyl ethyl ketone, and methyl isobutyl ketone), particulate matter, particulate trace metals, carbon dioxide, hydrogen chloride, sulfur dioxide, and nitrogen oxides. Only volatile POHCs were measured, thus there was no need for a modified EPA Method 5 sampling train. In addition, the distribution of the metals and chlorine were measured in all of the process input and output streams, i.e., the coal feed, waste fuel feed, clay feed, scrubber influent water, aggregate product, and scrubber effluent water. Waste fuel and coal samples were submitted for analyses of sulfur, ash, and Btu content. Waste fuel and scrubber effluent water also were analyzed for principal organics. Table 3-1 summarizes the overall test program and lists each sampling and analytical method used. Determination of carbon monoxide and products of incomplete combustion (PICs) in the stack gas was not performed.

Sampling was conducted for five full test days during the week of February 20-25, 1983. Because the kiln rarely operates on 100% coal fuel feed, the testing was conducted on normal fuel operation (i.e. coal and waste fuel) and no baseline (coal only) testing was performed.

TABLE 3-1. SUMMARY OF FLORIDA SOLITE AGGREGATE KILN SAMPLING AND ANALYTICAL PROGRAM

Parameter	Sampling method	Analytical method
Stack Gas		
· POHCs (tetrachloroethylene, toluene, MEK, MIBK)	Volatile organic sampling train (VOST)	GC/MS, thermal desorption and SIM
· Particulate matter	EPA 5	EPA 5
Metals on particulate	EPA 5	ICP
· Hydrogen chloride	Impinger absorption in 0.5 m NaOAc (back half of EPA 5)	Specific ion electrode
· CO_2 and O_2	EPA 3	Fyrite
· Nitrogen oxides	EPA 7	EPA 7
· Sulfur dioxide	EPA 6	EPA 6
Waste Fuel		
· Principal organics	Grab → composite	GC/MS
· Metals	Grab → composite	ICP
· Chlorine, sulfur	Grab → composite	XRF
· Btu content	Grab → composite	ASTM D240-64
· Ash content	Grab → composite	ASTM D482-IP4
Scrubber Discharge[a]		
· POHCs	Grab → composite	GC/MS
· Metals	Grab → composite	ICP
· Lead	Grab → composite	AAS
· Hexavalent chromium	Grab → composite	APHA312B
· Chlorine	Grab → composite	XRF
Aggregate Product		
· Metals	Grab → composite	ICP
· Chlorine	Grab → composite	XRF
Clay Feed		
· Metals	Grab → composite	ICP
· Chlorine	Grab → composite	XRF
Coal		
· Metals	Grab → composite	ICP
· Chlorine, sulfur	Grab → composite	XRF
· Btu and ash content	Grab → composite	ASTM D240-64
Scrubber Influent		
· Metals	Grab → composite	ICP
· Chlorine	Grab → composite	XRF

[a]The scrubber discharge was split into sludge and supernatant fractions and was analyzed separately where applicable.

3.1 LOCATION OF SAMPLING POINTS

The primary sampling site was the exhaust stack of the aggregate kiln after the air pollution control device, as shown in Figure 3-1. The stack is 27.4 m (90 ft) high and 0.9 m (3 ft) in diameter with a sampling platform 10.3 m (34 ft) high serving two 10.2 cm (4-in.) sampling ports. The ports are located 11.3 diameters from the nearest upstream disturbance (the junction of ductwork from the scrubber water knockout chamber) and 17.7 diameters from the nearest downstream disturbance (stack outlet). In accordance with EPA Method 1 criteria, a 12-point traverse (six points in each of the two ports 90° apart) was used for EPA Method 5 particulate test runs at 5 minutes per point. These ports also were used for EPA Methods 6 and 7 testing for SO_2 and NO_x, respectively, as well as for the volatile organic sampling train (VOST) for POHCs.

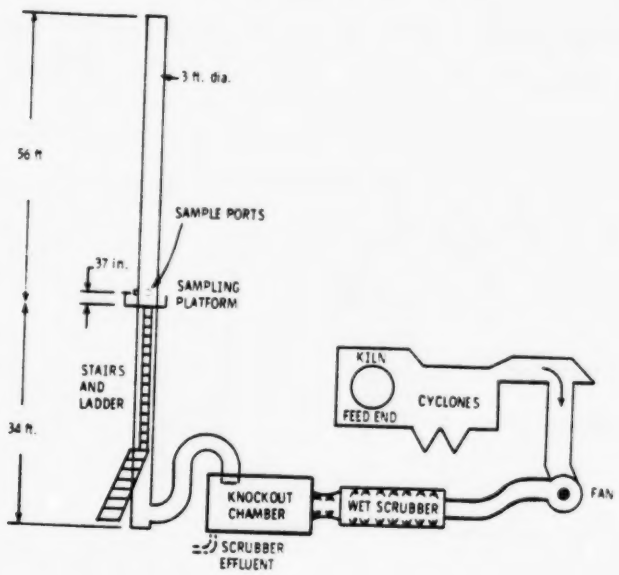

Figure 3-1. Air pollution control system and stack sampling site.

Figure 3-2 illustrates the sampling platform area which was slightly modified by the removal and welding of a portion of platform handrails to accomodate equipment movement.

The laboratory trailer was located at the base of the stack and contained EPA Method 5 and 6 and VOST console boxes, EPA Method 7 (NO_x) preparation area, EPA Method 5 clean-up space, miscellaneous lab bench space, and all sampling supplies. A Method 5 umbilical gasline was run from the trailer to the sampling platform along with Teflon lines for EPA Method 6 and VOST systems.

Other sampling locations used for collecting process samples included a valve in the waste fuel feed pipe near the kiln burner area, the crushed coal conveyor, the belt conveyor carrying clay feed to the kiln, the loadout pile for the expanded aggregate product, a valve in the plant water line leading to the scrubber, and the scrubber discharge pipe in the ditch leading to the scrubber holding pond. Sampling locations are marked with an asterisk in Figure 2-1. Several of the process sampling locations are shown in Figures 3-3 through 3-5. The process sites were sampled by compositing four grab samples per day into a daily process sample.

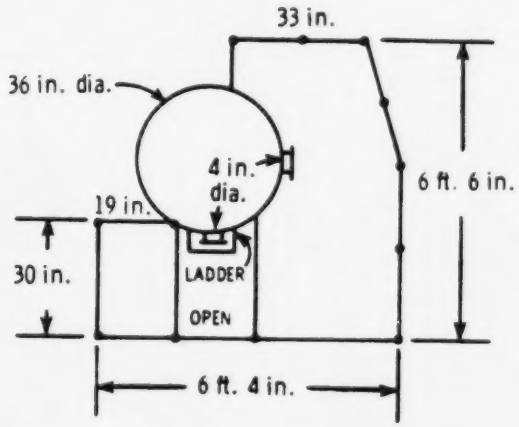

Figure 3-2. Sampling platform of kiln No. 5.

Figure 3-3. Clay feed belt to Kiln No. 5.

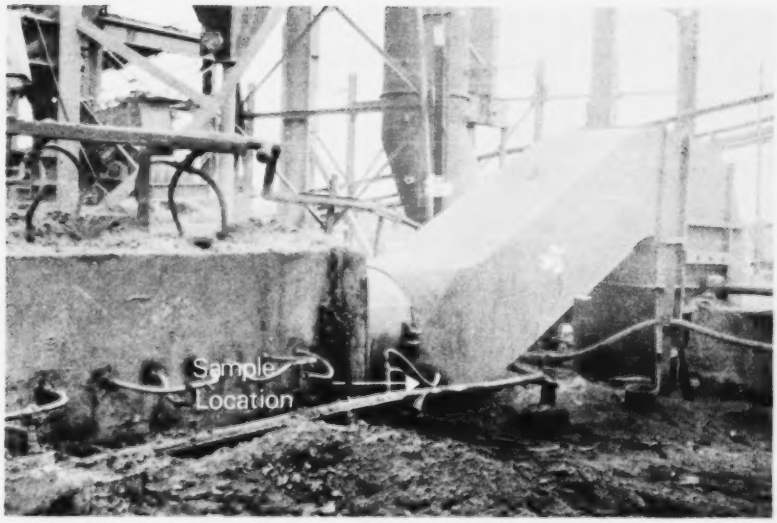

Figure 3-4. Duct work (right), scrubber system (left), and scrubber influent sample point (front).

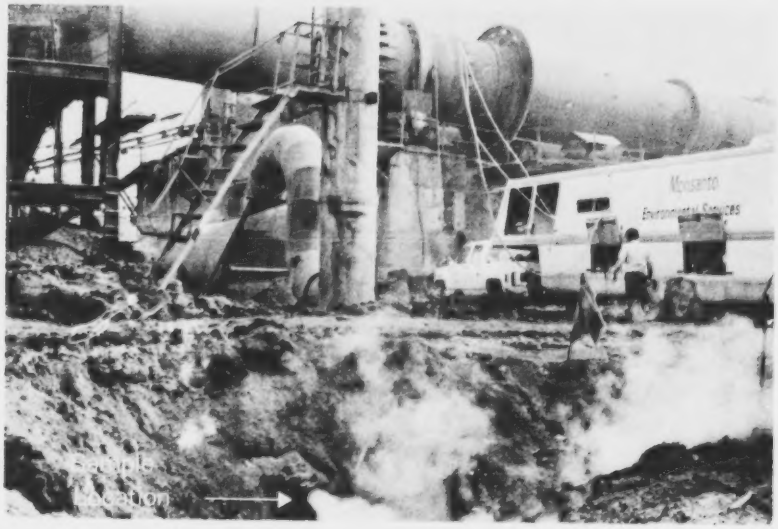

Figure 3-5. Scrubber discharge pipe (front, bottom), stack (middle), Kiln No. 5 (rear), and MRC sampling trailer (right).

intervals and composited to yield a single 500-mL sample. Samples were contained in a glass bottle with a Teflon-lined cap.

The waste fuel flow rate was determined daily at the plant by recording the difference in storage tank levels. Flowmeters were not installed or available at the plant due to the short operating life of the flowmeter internals and the inability to obtain vendor warrantees.

Principal organic components were analyzed using a Hewlett-Packard 5985 mass spectrometer, interfaced to a Hewlett-Packard 5840A gas chromatograph (GC/MC) with a 30-m fused silica DB 5 capillary column operated in the split injection mode. The column was programmed at 4°C/min from -10°C to 62°C for the analysis of low-boiling components, and at 8°C/min from 20°C to 300°C for the analysis of high-boiling components. The waste fuel samples were diluted 1:10 in o-xylene or methylene chloride before introduction into the GC/MS and analyzed in duplicate. Identification was

boiling components, and at 8°C/min from 20°C to 300°C for the analysis of high-boiling components. The waste fuel samples were diluted 1:10 in o-xylene or methylene chloride before introduction into the GC/MS and analyzed in duplicate. Identification was based on the mass spectra (MS) alone or on mass spectra and retention time. Each identified component was quantified relative to a standard of known concentration of that component injected into the GC/MS (major componets defined as >1% by weight), or semi-quantified relative to another compound which eluted in the vicinity of that compound (minor components defined as <1%). If a POHC was present at levels less than 1% in the waste, it was quantified against the external standard of the POHC, same as the major components.

The POHCs chosen for this program and analyzed by GC/MS, split injection mode for all waste fuel samples are tetrachloroethylene (Perc), toluene, methyl ethyl ketone (MEK), and methyl isobutyl ketone (MIBK). A wide-scan analysis to determine the top 20 components in the waste fuel also was conducted by GC/MS for only two fuel samples taken on Days 1 and 4.

Chlorine and sulfur levels were determined by x-ray fluorescence (XRF). The Btu content of the waste fuel samples was determined by ASTM 240-64, which is a Parr bomb technique. Ash content of the waste fuel samples was determined by ASTM D482-IP4, and specific gravity was obtained by the calibrated dilatometer method. Trace metal content of the waste fuel samples was determined by EPA Interim Method 200.7, modified by dilution in xylene followed by ICAP analysis. The ICAP method is an analytical system for simultaneous multielement determinations of trace metals at the sub-ppm levels in solutions. The ICAP analysis of waste fuels used for quality assurance (QA) purposes a standard containing metals of interest and known concentration in oil.

PCB concentration was determined by gas chromatography electron capture detector (GC/EC). Samples were initially extracted at a

neutral pH with methylene chloride, injected into the GC using the solvent flush technique, and detected by EC detector.

3.3 STACK SAMPLING AND ANALYSIS PROCEDURES

3.3.1 POHCs (W_{out})

POHCs in this program are volatile organic compounds. Collection on an adsorbent resin followed by thermal desorption into a GC/MS system was used to assure enough sample collection to demonstrate destruction and removal efficiencies (DRE).

The EPA Volatile Organic Sampling Train (VOST), shown schematically in Figure 3-6, utilizes a packaged tube of Tenax GC adsorbent resin for primary collection of the volatile organic species. Another packed tube also containing Tenax GC is placed downstream in the train as a backup to collect any POHC that break through from the primary collector. The sampling rate was 1 L/min, and the run time per sample averaged 20 minutes. A new pair of Tenax tubes was used after every 20 L of gas sampled. The tubes were changed after every 20 L of gas sampled for the following reasons:

- At sample volumes of greater than 20 liters, some of the very volatile POHCs will break through both the front and backup adsorbent tubes [1].

- Changing of the tubes allows an initial analysis of one pair of tubes. Analysis of a single pair of tubes lowers the possibility of collecting too much sample and overloading the GC/MS system. However, if the POHCs are not detected or are present at low levels in the single pair, the option

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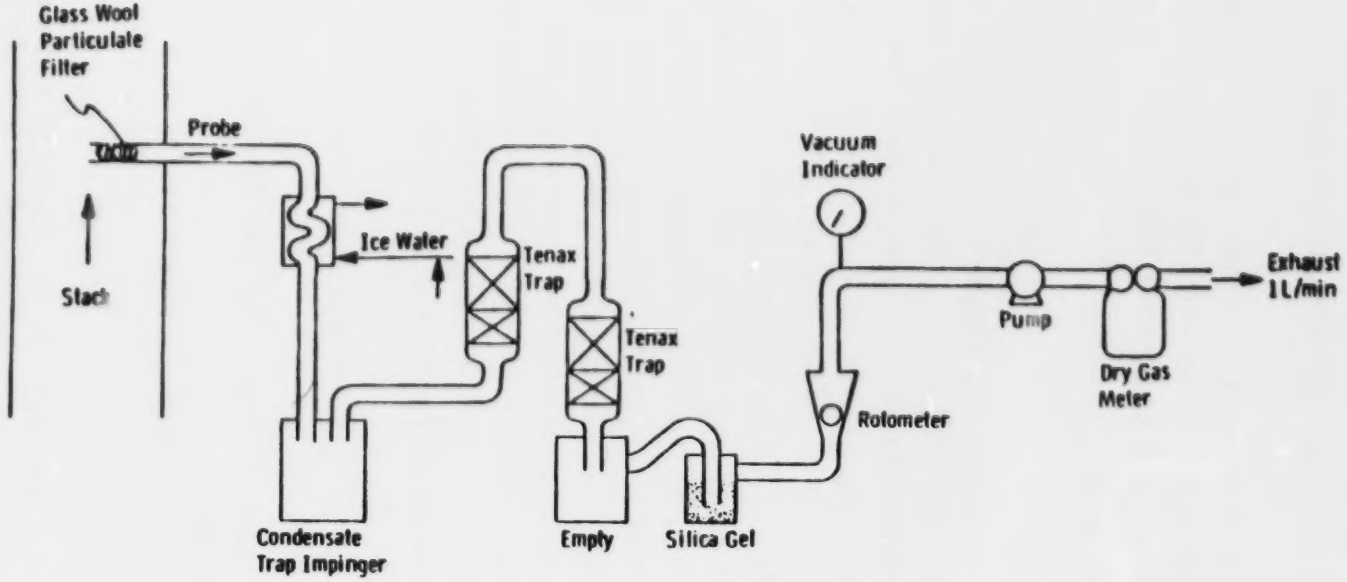
[1] Krost, K. J. and E. D. Pellizzari. "Collection and Analyses of Hazardous Organic Emissions." Analytical Chemistry 1982, 54, 810-817.



Note: Tenax traps are 1.6 cm in diameter and 10 cm long.
Tenax traps are mounted vertically.

Figure 3-6. Volatile organic sampling train (VOST).

exists of combining the contents of the remaining pairs of tubes by thermally desorbing one or more tubes onto one pair of tubes with a concomitant increase in sensitivity.

The required sample volume of 20 L was calculated prior to the test [2] to be within the range of the minimum sample gas volume needed to demonstrate best-case DRE (4 or 5 nines) and the maximum sample gas volume to avoid breakthrough of organic compounds.

The Tenax GC (35/60 mesh) was prepared by Soxhlet extraction for 24 hrs with methanol/hexane (1:1). The sorbent was then dried in a vacuum oven at 110°C for 6 hrs, and packed into 10-cm x 1.6-cm glass tubes. A minimum amount of pre-extracted and oven-dried glass wool was used to hold the sorbent in each of the tubes. The all-Tenax tube contained about 1.2 g of Tenax. The tubes were thermally conditioned prior to use, using a Nutech Model 322 thermal conditioning unit, with nitrogen at 200°C for 16 hrs. The conditioning gas was purified by passing through a U-trap containing a 5-Angstrom molecular sieve with the U-trap immersed in liquid nitrogen. The flow rate of gas through each trap was about 30 mL/min. Following conditioning, each tube, engraved with a unique number, was capped with Teflon caps and transferred to a clean 25-mm x 150-mm screw-cap culture tube ready for use.

The VOST train, shown in Figure 3-6, consisted of:

- A sampling line (1/4-in. Teflon tubing)
- First condenser
- Impinger (for condensate removal)

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[2] Peters, J. A., and Day, D. R. Quality Assurance Plan for Hazardous Waste Incineration Study, Florida Solite Aggregate Kiln. Contract 68-03-3025 SDM-06. U.S. Environmental Protection Agency, Cincinnati, Ohio, March 11, 1983.

- Tenax tube
- Tenax tube
- Empty impinger
- Silica gel
- Other sampling components (rotameter, pump, dry gas meter, greased stop cock)

Before each run, the tubes were connected in the sampling train, and the train was leak checked in accordance with the EPA Method 6 procedure. A leakage rate of $\leq$2% of the average sampling rate was acceptable. Small sealed charcoal tubes were used during leak checks to prevent contamination of the Tenax tubes by organic vapors in the ambient air. The probe of the train was then placed in the stack, and the train was operated for 20 minutes at 1 L/min gas sampling rate (gas flow rates in L/min refers to normal conditions of 20°C, 1 atm, dry basis). Multiple field and trip blanks* were taken to check for contamination of samples.

During each run, ice water was circulated through the condensers. After about 20 L of gas sample, a post-test leak check was performed. Two trains were built on one ring stand so that "back-to-back" runs were made. After the first run was completed, the second train was leak checked before starting the next 20-min sampling period. Upon use of the second train, a post-leak check was performed, and the two trains were removed from the stack. When each train was not in use the tubes were capped off with Swagelok®

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*A field blank is a pair of tubes installed in the VOST train, hoisted up the stack to the sampling location, retained at the stack for approximately 30 minutes without sampling, leak checked, hoisted down the stack, and removed from the train. A trip blank is not opened or removed from the shipping container except to be labelled.

type Teflon caps. The two trains were carried to the clean-up area in the site offices, where the used tubes were removed, placed into marked culture tubes, and replaced with new tubes.

GC/MS analysis for the four POHCs on the VOST tubes involved five basic steps listed below and described in detail in the following pargraphs.

- Spiking of the tubes with an internal standard (benzene d_6)
- Thermal desorption of the tubes
- Gas chromatography of the desorbed species
- Selected-ion monitoring (SIM) analysis of the POHCs eluting from the chromatographic column (with computer data storage)
- Recall of stored data and calulation of results.

Sample tubes, along with appropriate standards and laboratory blanks, were spiked immediately prior to analysis with 10 ng (10 µg/mL of a 1 µg/mL solution in methanol) of a benzene-d_6 internal standard. The tubes were then heated to 175°C for 15 minutes to desorb components onto a 6-ft x 1/4-in. S.S., 1% SP-100 on 60/80 Carbopack B GC column held at -40°C. The column was programmed to heat at 8°C/min. to 225°C for 7 minutes. The eluting components were analyzed using a Hewlett-Packard 5983 mass spectrometer operated in the selected ion monitoring mode which scans the major ions of a compound during a specified time frame. Data was collected using a Hewlett-Packard 5937 data system. Four major ions of each analyte and internal standard were monitored. External standards of the compounds of interest were used to determine the

correct ion ratios and compound retention times. These standards were prepared at two concentrations by spiking standard solutions of the analytes in methanol onto blank tubes, and then immediately desorbing in the same manner as the samples.

Concentrations of the analytes were calculated using the internal standard method and the following equation:

$$\text{Amount of analyte} = \frac{\text{AA (samp)}}{\text{AA (std)}} \times \frac{\text{AIS (std)}}{\text{AIS (samp)}} \times \text{AMT (std)}$$

where AA (samp) = area of analyte in the sample
AA (std) = area of analyte in the external standard
AIS (std) = area of internal standard in the standard run
AIS (samp) = area of internal standard in the sample run
AMT (std) = amount of analyte in external standard (wt)

The mass of a specific compound was then used along with sample meter conditions (i.e., sample volume and temperature) and stack conditions (i.e., stack flow rate) to calculate the mass emmission rate for a given compound.

3.3.2 Stack Gas Velocity, Flow Rate, Dry Molecular Weight and Moisture Content

The procedure and equipment used to determine stack gas conditions followed the specifications in EPA Methods 1 through 4 outlined in the Federal Register. The location of the traverse points (Method 1) was determined during setup days before testing. In addition, a preliminary velocity traverse (Method 2) was performed to obtain a preliminary stack velocity. Method 2 also was performed each day. Methods 3 and 4 were performed only during Method 5 runs. CO_2 and O_2 concentrations and dry molecular weight calculations (Method 3) were determined using Fyrite analyzers. No measurements were made

for carbon monoxide concentration. Method 5 back-half impingers were used for stack moisture determination (Method 4).

3.3.3 Particulate Matter

The procedure and equipment used for quantitative particulate collection met the specifications outlined in EPA Method 5. The flue gas exhaust stack was traversed for 12 points (6 point each on two perpendicular diameters) at 5 min./point for representative sample collection. Mass emissions were collected over a period of about one hour from at least 35 dry standard cubic feet of exhaust gas. A sample gas volume of >30 dscf is required under NSPS for cement kilns.

A schematic diagram of the EPA Method 5 sampling train is shown in Figure 3-7. The front half of the train (filter and probe rinse) was used to determine particulate loading. The back half of the train (impinger section) was used for HCl sampling (see HCl procedure discussion).

The Method 5 train probe consisted of a 5-ft glass-lined probe. Reeve Angel Type 934 AH filters were used. A handheld computer program was used to set up the nomograph for calculating the K factor during Method 5 testing.

After sampling, the filters were desiccated and then re-weighed using a calibrated analytical balance. The weight of collected particulate matter was determined by subtracting the initial weight (before sampling) from the final weight (after sampling) of the filter. Several filters were used as QC blanks. The concentration of particulate matter in the stack gas for the samples collected was computer-calculated from recorded sampling and analytical data using an HP-85.

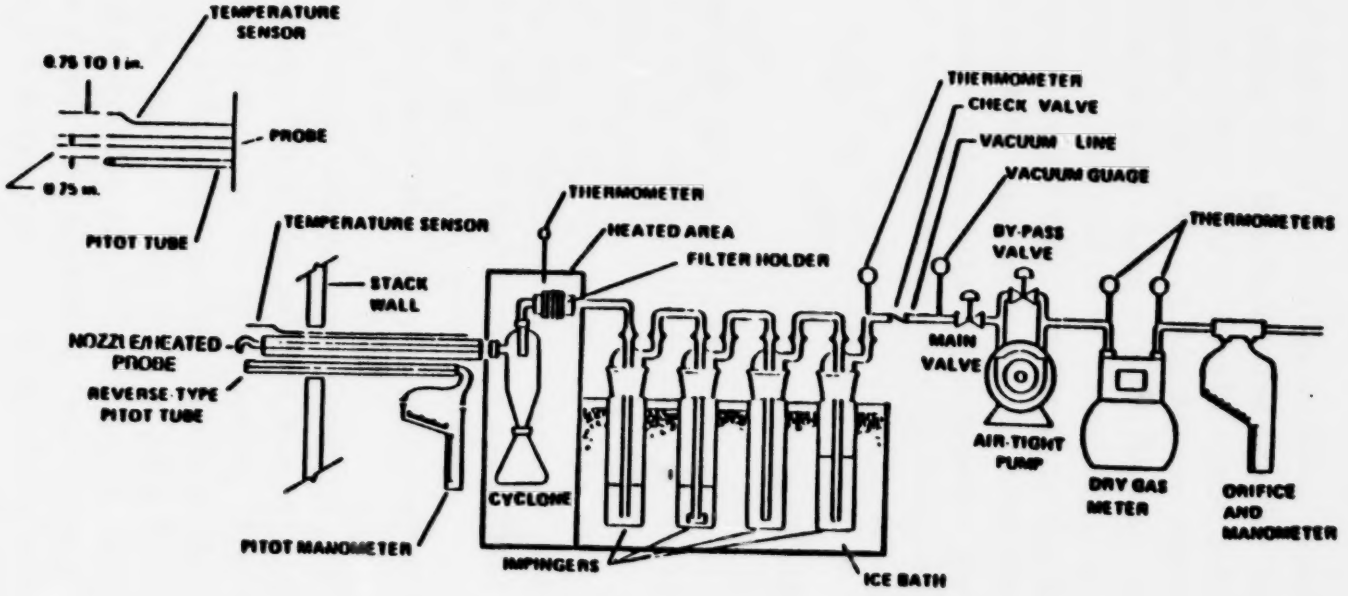

Figure 3-7. Particulate sampling train for EPA Method 5.

3.3.4 Metals on Particulate Matter

Particulate matter samples collected by Method 5 (evaporated probe rinse and filter) were analyzed for trace metals using inductively coupled argon plasma (ICAP) emission spectroscopy following EPA Interim Method 200.7. The ICAP method is an analytical system for simultaneous multielement determinations of trace metals at the sub-ppm level in solutions. The basis of the method is atomic emission promoted by coupling the sample, nebulized to form an aerosol, with high temperature argon plasma produced by passage of argon through a powerful radio frequency field. Blank filters were analyzed by ICAP to determine if any contamination existed due to the filter.

3.3.5 Hydrogen Chloride

HCl was collected in the back half of the EPA Method 5 sampling train in an absorbing collection medium of 0.5 M sodium acetate (NaoAc). The hydrogen chloride was trapped in the dilute solution, forming ionic sodium chloride. The sample was analyzed using a chloride specific ion electrode (SIE) and an expanded scale millivolt/pH meter. This sampling and analysis procedure is a modification of the NIOSH S246 procedure for HCl in air, scaled up for use on either midget or standard impinger sizes.

The impinger train, which was immersed in an ice bath during sampling, consisted of one bubbler and four impingers connected by glass U-tubes and clamps. Bubbler No. 1 and impinger No. 2 each contained 100 mL of sodium acetate absorbing solution. Impinger No. 3 contained 150 mL of 3% H_2O_2 solution, which removed SO_2 from the sample stream and protected the meter and pump. Impinger No. 4 was a dry impinger that functioned to remove moisture and/or catch carryover. Impinger No. 5 contained 200 to 300 g of 6 to 16-mesh silica gel.

For analysis, the collected sample was adjusted to a pH of 5 with acetic acid, and the chloride SIE reading was taken and compared to an analytical standards curve prepared from a series of five chloride standard solutions. Results were reported as μg Cl per sample, which are used to calculate stack gas concentration.

3.3.6 Sulfur Dioxide

Sulfur dioxide was collected and analyzed using EPA Method 6. Two 20-minute samples were collected each day using one midget bubbler, containing 15 mL of 80% isopropanol (IPA), and four midget impingers, two containing 15 mL of 30% H_2O_2, the third empty, and the fourth containing silica gel to protect the metering equipment. After the first day, an empty impinger was added to the train after the midget bubbler to catch carry-over caused by the high stack moisture. Each Method 6 sample consisted of at least 0.5 standard cubic feet of stack gas. Analytical results were used to calculate lb/SCF stack gas concentration following Method 6 procedures, and volumetric data from Methods 1 through 5 were used to calculate SO_2 mass emission rates (lb/hr).

3.3.7 Nitrogen Oxides

EPA Method 7 was used to determine emissions of nitrogen oxides. Four grab samples were collected over an approximately 1-hour period and analyzed individually; results were averaged to constitute one sample. Two samples (sets of four) were collected per day when possible. Sampling procedures included pipetting 25 mL of absorbing reagent (dilute H_2O_2/H_2SO_4) into ball flasks, evacuating the flasks to vacuum, drawing a stack gas sample into the flask, adding 1M NaOH to a pH of approximately 2, and recording ambient conditions, time of day, and flask pressures. Analyses were completed using the spectrophotometric acid procedure. Calculations were performed using an MRC computer program for reporting NO_x results in ppm or lb/hr of stack gas.

3.3.8 Process Samples

Coal, clay feed, aggregate product, scrubber influent and effluent, and waste fuel process samples were collected four times per day at approximately 2-hour intervals and composited to make one daily sample. Separate samples of scrubber effluent also were collected in 50-ml vials with no headspace and refrigerated for POHC analyses. Chlorine was determined in all samples using x-ray fluorescene (XRF), based on externally prepared standards. All process samples also were analyzed for trace metals by ICAP, using EPA Interim Method 200.7, as previously described. Coal samples were analyzed for sulfur, Btu, and ash content using ASTM standard methods. Scrubber discharge water also was analyzed for lead by atomic absorption, pH, and hexavalent chromium by the colorimetric method defined in Standard Methods 312 [3], and POHCs by purge/trap GC/MS protocol following EPA Method 624 [4]. Waste fuel samples were analyzed for POHCs, wide-scan organics, metals, chlorine, sulfur, Btu, ash, and specific gravity as previously described in Section 3.2. Scrubber effluent samples were also analyzed for EP toxicity (metals only) following methods specified in the Federal Register [5].

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[3] Standard Methods for Examination of Water and Wastewater. American Public Health Association, American Water Works Association, and the Water Pollution Control Federation. 15th edition.

[4] EPA Guidelines Establishing Test Procedures for the Analysis of Pollutants. Proposed Regulations. Federal Register. December 3, 1979.

[5] EP Toxicity Test Procedures - Appendix II. Code of Federal Register part 261.

SECTION 4

DEMONSTRATION TEST BURN RESULTS

4.1 TEST BURN RUNS

Sampling was conducted over a 5-day period, each day representing a run. Thus, Run No. 1 was conducted on Monday, Run No. 2 on Tuesday, etc. Multiple samples taken during the same run are designated by letters following the run number (i.e., Run No. 2B represents the second day, second sample taken).

The kiln operated normally, with slight variation in waste fuel rates and Btu input, except on Wednesday morning when the kiln was shut down due to a bearing failure on the I.D. fan. No samples were taken during the shutdown period, and several samples were eliminated to keep the sampling on schedule (as discussed in the following subsections).

4.2 STACK SAMPLING RESULTS

4.2.1 Stack Conditions

Stack conditions as measured by EPA Methods 1 through 4 during Method 5 sampling are shown in Table 4-1. The stack was saturated with moisture at approximately 70°C (158°F) due to the water scrubber particulate removal system. Stack velocities averaged 16.8 m/sec (55.2 ft/sec) and were very consistent over the week-long test period. No EPA Method 5 run was made on Tuesday (Run No. 2) so that additional VOST runs could be performed.

TABLE 4-1. STACK CONDITIONS, KILN NO. 5 FLORIDA SOLITE CORPORATION, FEBRUARY 21-25, 1983.

Run number	Date	Stack temperature °C	Stack temperature °F	Stack velocity m/sec	Stack velocity ft/sec	Stack static pressure cm H_2O	Stack static pressure in. H_2O	Water content, %	Percent CO_2	Percent O_2	Dry molecular weight, kg/kg-mole (lb/lb-mole)	Stack flow rate, dry dscm/min	Stack flow rate, dry dscf/min	Stack flow rate, actual m^3/min	Stack flow rate, actual ft^3/min
1	2/21/83	67	153	16.8	55.2	-2.2	-0.85	21.5	1.7	13.9	29.58	454	16,030	661	23,350
2	2/22/83	68	154	17.1	56.2	-2.2	-0.85	NA[a]	NA[a]	NA[a]	NA[a]	428	15,130[b]	673	23,770
3	2/23/83	71	160	16.6	54.6	-2.2	-0.85	26.4	0.7	14.8	29.64	410	14,470	654	23,090
4	2/24/83	73	163	16.9	55.4	-2.2	-0.85	28.8	2.4	12.2	29.67	401	14,150	623	23,400
5	2/25/83	72	161	16.6	54.4	-2.2	-0.85	27.9	1.3	13.7	29.67	400	14,110	651	22,990

[a]No EPA Method 3, 4 or 5 runs were made.

[b]Day 3 moisture and molecular weight data were used.

4.2.2 Particulate and HCl Emissions

Particulate emission rates remained at relatively low levels for all tests averaging 5.3 kg/hr (11.7 lbs/hr) as shown in Table 4-2. No runs exceeded the current air permit regulations for this site of 8.82 kg/hr (19.43 lb/hr) issued by the Florida Department of Environmental Regulations (DER). These values also agree with previous testing results conducted one month earlier at the site which averaged 6.1 kg/hr (13.40 lb/hr) of particulate emissions. No EPA Method 5 run was made on Day 2 so that additional VOST runs could be performed.

The average particulate stack gas concentration was 214.7 mg/dscm ± 96.7 mg/dscm (95% confidence limits) (0.094 grains/dscf ± 0.021 grains/dscf).

HCl emissions averaged 0.023 kg/hr ± 0.020 kg/hr (95% confidence limits) (0.051 lb/hr ± 0.044 lb/hr). On Day 1, the first particulate run was unacceptable owing to a cracked filter; however, the back half of the Method 5 train was valid for HCl. Combined with the accepted particulate Run 1, there are two HCl emission rates averaged for Run 1.

Isokinetics ranged from 91% to 95% for all EPA Method 5 runs, within the 100% ± 10% acceptable range prescribed by the Federal Register.

4.2.3 Metals on Particulates

Results of trace metals analysis by ICAP on particulate filters and probe washes are shown in Table 4-3. If the results for filters and probe washes are combined to obtain a single value for each EPA Method 5 run, the trace metals of highest concentration include sodium (35,000 to 52,000 µg/g), lead (32,000 to 52,000

TABLE 4-2. PARTICULATE AND HCl STACK GAS CONCENTRATIONS, KILN NO. 5 FLORIDA SOLITE CORPORATION, FEBRUARY 21-25, 1983.

Run number	Sample duration, min	Sample volume		Particulate concentration		Particulate emission rate		HCl concentration, ppm	HCl emission rate		Percent isokinetic
		dscm	dscf	mg/dscm	grains/dscf	kg/hr	lb/hr		kg/hr	lb/hr	
1	70	1.41	49.0	162.6	0.071	4.4	9.7	0.45[a]	0.024[a]	0.052[a]	93.6
2	NA[b]	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
3	60	1.05	37.1	233.6	0.102	5.8	12.7	0.15	0.008	0.017	91.6
4	60	1.06	37.5	272.5	0.119	6.5	14.4	0.68	0.034	0.076	94.5
5	60	1.04	36.7	190.1	0.083	4.5	10.0	0.54	0.027	0.060	93.0

[a]Average of two values from Runs 1 and 1A.

[b]NA - data not available; EPA Method 5 run was not made.

TABLE 4-3. TRACE METAL RESULTS FOR EPA METHOD 5 SAMPLES, KILN NO. 5 FLORIDA SOLITE CORPORATION, FEBRUARY 21-25, 1983.

Metal	Filter concentration, µg/gram[a]				Probe wash concentration, µg/gram[a]			
	1	3	4	5	1	3	4	5
Aluminum	18,500	21,200	22,600	22,300	19,600	20,800	21,100	19,300
Boron	86.8	73.6	196	248	127	105	89.1	138
Barium	626	757	865	1,320	110	161	124	187
Beryllium	4.4	5.0	5.3	4.5	2.0	1.7	2.0	1.3
Calcium	15,300	18,300	21,100	14,600	6,920	7,470	8,780	8,710
Cadmium	439	487	488	274	55.9	74.3	69.0	53.0
Cobalt	46.0	47.3	55.9	48.6	40.9	29.6	45.4	30.4
Chromium	421	322	337	731	839	1,240	1,030	523
Copper	953	923	925	1,080	256	348	286	318
Iron	14,900	17,000	17,600	18,200	16,800	19,300	17,700	17,700
Magnesium	2,210	2,520	2,890	2,860	7,450	7,710	8,280	6,890
Manganese	122	153	209	154	225	262	271	170
Molybdenum	699	412	421	1,540	90.8	126	115	89.4
Sodium	35,400	26,100	28,800	17,300	17,000	18,200	16,500	18,900
Nickel	55.9	56.5	50.6	63.0	1,100	753	1,250	540
Lead	29,700	31,000	31,900	48,800	2,690	1,970	2,040	3,190
Tin	ND[b]	ND	ND	ND	ND	ND	ND	ND
Titanium	641	993	1,060	1,150	761	944	886	1,010
Vanadium	135	164	134	145	30.8	44.9	53.1	21.2
Yttrium	23.7	15.6	24.9	18.1	15.8	17.9	21.6	17.1
Zinc	5,230	5,690	5,940	3,580	1,350	1,420	1,440	1,140

[a] No EPA Method 5 run was made on Day 2.

[b] ND - not detected (or ≤ dilution factor x detection limits in last column of Table 4-11); dilution factors are as follows: Filter 1 624, Wash 1 431; Filter 3 742, Wash 3 467; Filter 4 693, Wash 4 347; Filter 5 884, Wash 5 589

µg/g), aluminum (38,000 to 43,000 µg/g), iron (32,000 to 36,000 µg/g), calcium (21,000 to 30,000 µg/g), magnesium (9,000 to 11,000 µg/g), zinc (5,000 to 7,400 µg/g), and chromium, copper, and titanium (1,200 to 2,100 µg/g). All other ICP trace metals are less than 1,500 µg/g. The data is consistent between days with relatively small fluctuations in trace metal concentrations. Filter, probe (acetone) wash, and lab reagent blanks were prepared and analyzed along with the filters and probe washes for QA/QC. All QA/QC data and results are presented in Section 7.

Mass emission rates for the trace metals are shown in Table 4-4 and were calculated by the following equation:

$$M_m = M_s \times C_m \times \frac{1}{1,000} \qquad (4\text{-}1)$$

where:

M_m = mass emission rate of a trace metal, g/hr

M_s = mass rate of particulates from stack, kg/hr (from Table 4-2)

C_m = concentration of a trace metal, µg/gram (from Table 4-3)

$\frac{1}{1,000}$ = conversion factor to correct units

Trace metals having highest emission rates include sodium (163 to 305 g/hr), aluminum (168 to 284 g/hr), lead (142 to 234 g/hr), iron (139 to 229 g/hr), calcium (98 to 194 g/hr), magnesium (42 to 739 g/hr), and zinc (21 to 48 g/hr). All other ICP trace metals had emission rates less than 10 g/hr.

4.2.4 Sulfur Dioxide Emissions

Emmissions of SO_2 ranged from 72.2 kg/hr to 99.6 kg/hr (159.1 lb/hr to 219.4 lb/hr) and averaged 82.4 kg/hr (181.4 lb/hr) as

TABLE 4-4. MASS EMISSION RATE FOR TRACE METALS FROM STACK GAS

Metal	Emission rate, g/hr			
	1	3	4	5
Aluminum	168	244	284	187
Boron	0.94	1.0	1.9	1.7
Barium	3.2	5.3	6.4	6.8
Beryllium	0.03	0.04	0.05	0.03
Calcium	97.7	149	194	105
Cadmium	2.2	3.3	3.6	1.5
Cobalt	0.38	0.44	0.66	0.36
Chromium	5.5	9.1	8.9	5.6
Copper	5.3	7.4	7.9	6.3
Iron	139	211	229	162
Magnesium	42.5	59.3	72.6	43.9
Manganese	1.5	2.4	3.1	1.5
Molybdenum	3.5	3.1	3.5	7.3
Sodium	230	257	305	163
Nickel	5.1	4.7	8.5	2.7
Lead	142	191	221	234
Tin	ND[a]	ND	ND	ND
Titanium	6.2	11.2	12.6	9.7
Vanadium	0.73	1.2	1.2	0.75
Yttrium	0.17	0.19	0.30	0.16
Zinc	28.9	41.2	48.0	21.2

[a]Not detected.

shown in Table 4-5. A Dixon test [6] on Run No. 1A shows that the 42.3 lb/hr value is an outlier and is statiscally eliminated from analysis of the data. SO_2 stack gas concentrations ranged from 2,731 mg/m^3 to 3,924 mg/m^3, and averaged 3,346 mg/m^3 ± 860 mg/m^3 (95% confidence limits) (1,257 ppm ± 323 ppm). No EPA Method 6 runs were made on Day 3 because the plant was shut down in the morning and no time was available in the afternoon because particulate, VOST, and NO_x runs were being made.

4.2.5 Nitrogen Oxides Emissions

Emissions of NO_x averaged 7.7 kg/hr (17 lb/hr) and ranged from 1.9 kg/hr to 11.7 kg/hr (4.1 lb/hr to 25.8 lb/hr) as included in Table 4-5. Only one set of EPA Method 7 runs were made on Day 3 during the afternoon because the plant was shut down in the morning. NO_x (as NO_2) stack gas concentrations averaged 304 mg/m^3 ± 251 mg/m^3 (95% confidence limits) (162 ppm ± 133 ppm). The NO_x sample in Run 3A was taken during kiln start-up after the morning shutdown and thus has an expected low value of 39.5 ppm.

4.2.6 POHC Emissions (W_{out})

Six volatile organic sample train (VOST) runs were scheduled per day. However, initial problems on Day 1 with leak checking the VOST train and excessive moisture plugging of the Tenax tubes resulted in a low number of samples being taken on Day 1 and extra samplings being taken on subsequent days as follows:

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[6] Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I Principles. EPA 600/9-76-005, U.S. Environmental Protection Agency, Research Triangle Park, NC, 1976.

TABLE 4-5. SULFUR DIOXIDE AND NITROGEN OXIDES STACK GAS CONCENTRATIONS

Run number	SO_2 emissions rate		SO_2 concentration		NO_x emissions rate		NO_x concentration	
	kg/hr	lb/hr	mg/m^3	ppm	kg/hr	lb/hr	mg/m^3	ppm
1A	19.2	42.3	717	269.6	8.5	18.7	312.6	164.2
1B	73.2	161.2	2,731	1,026	11.7	25.8	431.2	227.0
2A	99.6	219.4	3,924	1,474	10.9	24.1	426.4	224.6
2B	88.0	193.8	3,466	1,302	3.5	7.8	136.3	72.2
3A	-[a]	-[a]	-[a]	-[a]	1.9[b]	4.1[b]	75.3[b]	39.5[b]
3B	-[a]	-[a]	-[a]	-[a]	-[b]	-[b]	-[b]	-[b]
4A	75.3	165.8	3,173	1,192	6.5	14.4	272.5	143.7
4B	77.8	171.4	3,290	1,236	7.7	17.0	322.2	169.6
5A	90.4	199.0	3,831	1,439	9.0	19.9	378.3	199.3
5B	72.2	159.1	3,008	1,130	9.7	21.4	405.6	213.6

[a] Plant down; no SO_2 runs made on Day 3.

[b] Plant down in morning; only 1 NO_x run made on Day 3.

Day No.	No. of VOST runs made
1	1
2	9
3	6
4	8
5	6

Leak check problems voided several runs on Day 1. Leak check problems were caused by (1) unseen cracks in the silica gel drying tube, which was corrected by putting the silica gel in a midget impinger, or (2) grit between the impinger glass ball connectors, which required periodic cleaning of the connectors. Excessive moisture caused plugging in the Tenax tubes and voided one run on Day 1 and two runs on Day 2. This was corrected by changing the VOST train configuration from probe-condenser-Tenax tube-impinger to the schematic shown earlier in Figure 3-6 of probe-condenser-impinger-Tenax tube after Run 2E. As a result of this change, approximately 5 ml of condensed moisture collected in the impinger per run instead of plugging the Tenax tube. The total nanograms (ng) collected for each POHC for each run, shown in Table 4-6 and calculated by the equation 4-2, does not include possible POHCs collected in the VOST impingers. This moisture is addressed in section 5.1.

$$\text{Total ng} = (\text{Front tube-Front tube blank}) + (\text{Back tube-Back tube blank}) \qquad (4\text{-}2)$$

Approximately one-half of the back tubes were initially analyzed and found to be less than 1.5 times the back tube field blanks and less than 10% the front tube mass. Thus, the second part of Equation 4-1 was assumed to be zero. Because the back tubes that had been analyzed were closely equal to the blank tubes, the remaining back tubes were not analyzed. Runs 1A, 2A, 2B, and 2D were voided during analyses owing to plugged Tenax tubes. Run 5D was not analyzed because of a cracked tube.

TABLE 4-6. TOTAL NANOGRAMS COLLECTED PER VOST RUN

Run number	Total nanograms collected			
	MEK[a]	MIBK[b]	Perc[c]	Toluene
1A	Plug[d]	Plug	Plug	Plug
2A	Plug	Plug	Plug	Plug
2B	Plug	Plug	Plug	Plug
2C	Void[e]	Void	Void	Void
2D	Plug	Plug	Plug	Plug
2E	Void	Void	Void	Void
2F	14.5	11.8	0[f]	0[f]
2G	9.0	17.7	7.2	112.3
2H	16[f,g]	20[f,g]	4.0	168.7
2I	0[f]	0[f]	0.5[f]	0.5[f]
2J	0[f]	1.2	2.7	94.7
2K	3.4[f]	5.6	4.1	82.0
3A	74[g]	209[g]	27[g]	290[g]
3A[a]	1,900[g]	140[g]	20[g]	690[g]
3B	135[g]	111[g]	15[g]	316[g]
3B[a]	4,760[g]	200[g]	15[g]	1,010[g]
3D	16.0[h]	88.8[h]	14.3[h]	574.8
3D[1]	123.8[h]	88.7[h]	16.1[h]	585.6
4A	337.4	926.4	25.1	>12,260.1
4B	222.3	552.3	15.8	1,541.3
4C	129.9	105.4	11.8	652.2
4D	142.7	280.4	11.4	965.6
4E	125.4	180.9	7.8	757.6
4F	142.5	151.8	6.7	112.7
4G	156.9	393.0	8.9	567.6
4H	61.8	201.4	6.7	680.7
5A	27.1	87.9	10.7	559.6
5B	34[f,2]	86.5	6.8	169.4
5C	0[f]	75.4	10.4	495.6
5D	Broke[i]	Broke	Broke	Broke
5E	159.5	176.3	18.1	1,013.5
5F	55.3	83.9	10.5	205.4

[a] Methyl ethyl ketone.

[b] Methyl isobutyl ketone.

[c] Tetrachloroethylene (Perc).

[d] Tubes plugged and not analyzed.

[e] Run voided in field due to plugging of tube.

[f] Mass on front tube not greater than two times blank tubes.

Blank values, ng: MEK = 8.72
MIBK = 0.92
Perc = 1.23
Toluene = 2.44

[g] Duplicate (side-by-side) sample sent to QA contractor.

[h] Duplicate (side-by-side) sample for internal MRC analysis.

[i] Sample tube was broken and not analyzed.

Runs 3A ($3A^3$) and 3B ($3B^3$) were side-by-side duplicate sampling trains that were split between MRC and the EPA Quality Assurance contractor. Run 3D ($3D^1$) was a side-by-side duplicate sampling train for MRC internal quality assurance practices. Results comparing these runs are presented in Section 7 of this report. Run 3C ($3C^3$) was a field blank also split with the EPA QA contractor. Results of all front, back, and blank tubes are provided in Appendix A.

Table 4-7 shows the mass emission rate for each POHC for each run (or W_{out} value). This rate will be used in Section 5 to calculate destruction and removal efficiencies (DRE).

4.3 PROCESS SAMPLE RESULTS

4.3.1 Waste Fuel (W_{in})

Results of waste fuel analyses for chlorine, sulfur, PCB, and other parameters are shown in Table 4-8. On Day 4 (~1100 hrs) the waste fuel feed supply was switched from Tank 5 to Tank 4. The characteristics of the new waste fuel supply were changed slightly as evidenced by the lower percent chlorine for Days 4 and 5. The waste fuel on Day 4 has a lower sulfur and heat value and a higher ash and specific gravity than that on other days. The difference from other samples is most likly attributable to pumping a settled slude material from the bottom of Tank 5 during the morning hours of Day 4 prior to the tank switch. The sludge material has a greater metals and inorganic concentration and thus a lower heat value and higher specific gravity than the non-settled liquid phase of the waste fuel. PCB concentrations were not detected (<0.1 ppm) for four of five samples. Sample No. 2 had a 10 ppm concentration. This sample was also analyzed a second time to verify the higher PCB concentration. The PCB limit allowed by the Florida DER permit for hazardous waste burning at Solite is 25 ppm.

TABLE 4-7. STACK EMISSION RATES FOR POHCs (W_{out})

Run number	Gas volume sampled, dscm[b]	Stack gas rate, dscm/min[c]	Stack emission rate (W_{out}), g/min x 10^{-3} [a]			
			MEK	MIBK	Perc	Toluene
2F	0.0199	428	0.313	0.254	0[d]	0[d]
2G	0.0195	428	0.198	0.389	0.158	2.47
2H	0.0195	428	0[d] 371	0[d] 439	0.089	3.71
2I	0.0196	428	0[d]	0[d]	0.012[d]	0.011[d]
2J	0.0198	428	0[d]	0.026	0.060	2.05
2K	0.0197	428	0.074	0.121	0.089	1.79
3A	0.0221	410	1.13	3.88	0.508	5.39
3A[3]	0.0209	410	37.3	2.75	0.393	13.6
3B	0.0237	410	2.35	1.92	0.259	5.48
3B[3]	0.0243	410	80.4	3.38	0.253	17.1
3D	0.0191	410	0.343	1.90	0.306	12.3
3D[1]	0.0201	410	2.53	1.81	0.328	12.0
4A	0.0203	401	6.67	18.3	0.496	>44.7
4B	0.0201	401	4.44	11.0	0.315	30.8
4C	0.0200	401	2.61	2.12	0.236	13.1
4D	0.0198	401	2.89	5.68	0.230	19.6
4E	0.0198	401	2.54	3.66	0.157	15.3
4F	0.0197	401	2.90	3.09	0.137	2.30
4G	0.0195	401	3.23	8.09	0.183	11.7
4H	0.0193	401	1.28	4.17	0.138	14.1
5A	0.0201	400	0.539	1.75	0.213	11.1
5B	0.0198	400	0[d] 688	1.74	0.136	3.41
5C	0.0198	400	0[d]	1.52	0.209	10.0
5E	0.0197	400	3.24	3.58	0.367	20.6
5F	0.0197	400	1.12	1.70	0.213	4.17

[a] Runs 1A, 2A through 2E, and 5D were voided in the field or during analyses and are not presented in this table.

[b] VOST sample volumes are presented in Appendix A.

[c] From EPA Method 5 sampling (see Table 4-1).

[d] Front tube mass less than two times blank values.

TABLE 4-8. WASTE FUEL CONDITIONS

Run number	Chlorine, %	Sulfur, %	PCB, ppm	Heat value, Btu/lb	Ash, %	Specific gravity, g/cc	Feed rate, gal/min	Mass rate, g/min
1	1.08	0.41	ND[a]	12,550	7.74	0.966	4.56	16,670
2	1.08	0.41	10	11,450	7.28	0.922	5.84	21,930
3	1.04	0.39	ND	12,740	7.47	0.978	3.74	13,850
4	0.55	0.26	ND	9,530	15.5	1.07	2.88	11,660
5	0.55	0.42	ND	12,670	6.18	0.966	3.63	13,270

[a]ND - not detected, detection limit = 0.1 ppm.

Two waste fuel samples from Days 1 and 4 were analyzed for the top twenty major components by capillary GC/MS, and results are shown in Table 4-9. As noted on the list, toluene and xylene are in highest concentration in the waste fuels. The sums of the concentrations listed represent only 35% to 40% of the total sample weight. About one-half of the samples was composed of a sludge-like precipitate, which was largely insoluble in organic solvents and would not be analyzed by GC/MS, thus accounting for the relatively small percentage of the sample weight analyzed. This sludge portion is largely inorganic in nature and has an extremely high metals content compared to the liquid portion.

Tetrachloroethylene is the only chlorinated compound listed in the top twenty compounds of Table 4-9, and it represents approximately 10% of the total chlorine in the sample. The remaining chlorine is most likely attributable to many, very low concentration organic chlorinated species and inorganic chlorides in the sludgy material.

The concentration of the POHCs in the waste fuel is shown in Table 4-10. All of the POHCs were relatively constant in concentration during the test except for tetrachloroethylene (Perc) which was reduced from approximately 0.18% to 0.05% between Days 3 and 4. This change was probably caused by the switch in waste fuel storage tanks which also is reflected in the percent chlorine shown in Table 4-8 and discussed previously.

The mass rates of each POHC for each day also are shown in Table 4-10. These values are the "W_{in}" used for the destruction and removal efficiency (DRE) calculations in Section 5.

Table 4-11 shows the concentrations of trace metals in the liquid and sludge phases of the waste fuel samples. The metals are concentrated in the sludge phase. Calcium and sodium are in the highest concentration, followed by lead, iron, aluminum, zinc, and magnesium.

TABLE 4-9. RESULTS OF CAPILLARY GC/MS ANALYSIS OF MAJOR COMPONENTS OF WASTE FUELS NUMBER 1 AND NUMBER 4

Waste fuel component	Concentration, wt %			
	Number 1	Number 1 duplicate	Number 4	Number 4 duplicate
Ethanol	1.62	1.49	1.77	1.88
2-Propanol	4.88	4.21	1.94	2.00
1-Butanol	1.73	1.82	0.666	0.873
Ethyl acetate	0.703	0.632	0.697	0.749
Methyl ethyl ketone[a]	2.02	2.05	2.52	3.09
Methyl isobutyl ketone[a]	1.54	1.51	1.09	1.15
Toluene[a]	8.28	8.52	7.85	8.26
Tetrachloroethylene[a]	0.190	0.200	0.068	0.072
Ethylbenzene	1.21	1.26	2.20	2.37
Xylene (isomer No. 1)	4.47	4.47	7.62	8.15
Styrene	0.680	0.730	0.279	0.278
Xylene (isomer No. 2)	1.30	1.29	2.42	2.62
2-Ethoxyethyl acetate	2.03	2.03	1.14	1.26
C_3-Benzene (isomer No. 1)[b]	0.470	0.470	0.325	0.342
C_3-Benzene (isomer No. 2)[b]	0.570	0.570	0.330	0.379
C_{10}-Alkane (isomer)[c]	0.840	0.820	0.740	0.786
C_{11}-Alkane (isomer)[d]	0.73	0.72	0.560	0.630
n-Propyl acetate	1.51	1.49	0.988	1.03
2-Propanol, 1-(2-methoxy-1-methylethoxy)-isomer No. 1	0.460	0.45	0.137	0.142
2-Propanol, 1-(2-methoxy-1-methylethoxy)-isomer No. 2	0.50	0.48	0.147	0.176
2-Cyclohex 4-1-one,trimethyl (isomer)	1.33	1.23	0.514	0.569

[a] POHC.

[b] Compounds containing three carbons associated with a benzene ring.

[c] Compounds containing ten carbons associated with an alkane.

[d] Compounds containing eleven carbons associated with an alkane.

TABLE 4-10. POHCs IN WASTE FUEL

POHC	Waste fuel concentration, %					Waste fuel mass rate (W_{in}), g/min				
	1	2	3	4	5	1	2	3	4	5
Methyl ethyl ketone (MEK)	1.99	1.78	1.83	2.81	4.25	331.7	390.4	253.5	327.7	564.0
Methyl isobutyl ketone (MIBK)	1.53	1.70	1.41	1.12	3.90	255.1	372.8	195.3	130.6	517.5
Tetrachloroethylene (Perc)	0.187	0.194	0.173	0.059	0.031	31.2	42.5	24.0	6.88	4.11
Toluene	8.38	9.27	8.21	7.99	7.54	1,397	2,033	1,137	931.6	1,000

TABLE 4-11. TRACE METALS IN LIQUID AND SLUDGE PHASE OF WASTE FUEL

Metal	Concentration, mg/L Waste fuel - liquid phase					Concentration, mg/L Waste fuel solid phase					Detection limit, ppm
	1[b]	2[b]	3[b]	4[a,b]	5[b]	1[c]	2[d]	3[e]	4[f]	5[g]	
Aluminum	ND[a]	ND	ND	ND	ND	3,821	3,770	3,725	4,312	5,796	0.06
Boron	6.6	6.9	7.2	5.5	6.4	46.2	51.2	49.2	59.1	65.2	0.02
Barium	ND	ND	ND	ND	ND	698	700	687	1,467	1,327	0.01
Beryllium	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.002
Calcium	231.9	228.5	203.3	224.5	276.7	33,960	28,470	25,150	20,610	15,000	0.025
Cadmium	1.5	1.5	1.9	ND	1.2	32.6	33.5	36.6	49.9	19.0	0.012
Cobalt	9.3	9.1	10.9	5.0	7.0	68.0	69.0	87.6	143	77.1	0.03
Chromium	2.7	2.7	3.0	1.1	3.2	733	827	897	718	1,730	0.01
Copper	16.2	15.4	17.5	17.8	27.7	186	200	295	497	327	0.02
Iron	8.6	8.4	9.1	4.9	7.5	3,977	3,984	4,488	9,080	6,393	0.045
Magnesium	12.8	12.7	12.2	9.9	12.3	1,595	1,358	1,326	1,880	1,257	0.03
Manganese	0.7	0.7	0.7	0.2	0.5	187	185	193	272	113	0.002
Molybdenum	ND	ND	ND	ND	ND	43.0	58	55.0	27.7	116	0.05
Sodium	541	614	760	216	133	13,190	14,330	18,420	12,690	14,750	6.0
Nickel	ND	ND	ND	ND	ND	34.2	34.0	36.9	48.1	40.0	0.065
Lead	15.1	14.5	19.0	10.3	90	3,932	4,416	4,903	5,320	7,781	0.095
Tin	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.55
Titanium	0.6	0.7	0.7	ND	0.5	323	286	310	252	188	0.01
Vanadium	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.03
Yttrium	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.025
Zinc	90.1	84.1	100.5	15.5	60.3	1,953	2,017	2,154	1,393	1,641	0.015

[a] Average of lab duplicates.

[b] ND ≤ 50 x detection limit in last column.

[c] ND ≤ 254 x detection limit in last column.

[d] ND ≤ 248 x detection limit in last column.

[e] ND ≤ 251 x detection limit in last column.

[f] ND ≤ 198 x detection limit in last column.

[g] ND ≤ 224 x detection limit in last column.

The detection limit for the waste fuel solid phase samples shown in Table 4-11 varies considerably. The detection limit was based on the dilution factor multiplied by the actual detection limit.

4.3.2 Coal

Results from the analyses of coal samples taken are shown in Table 4-12. The coal feed rate is an average value measured over a 1-week period provided by Florida Solite. Sulfur varied slightly from 2.86% to 3.86%; Btu content was consistent over the test period; chlorine content was less than the 50 µg/g detection limit.

Table 4-13 shows trace metal results in the coal.

4.3.3 Clay Feed, Aggregate Products, and Scrubber Influent

Results of chlorine analyses and mass rates for the clay feed, aggregate product, and scrubber influent water are shown in Table 4-14. Chlorine was not detected by x-ray fluoresence analysis in the clay and aggregate samples. Scrubber influent flow rate was

TABLE 4-12. COAL CONDITIONS

Run number	Sulfur, %	Ash, %	Heat value, Btu/lb	Chlorine, µg/g	Feed rate[b], lb/hr	Feed rate[b], kg/hr
1	3.37	9.06	12,720	ND[a]	1,540	700
2	3.54	7.78	12,830	ND	1,540	700
3	3.19	6.23	13,410	ND	1,540	700
4	2.86	8.09	13,070	ND	1,540	700
5	3.86	6.44	13,260	ND	1,540	700

[a]ND - not detected; detection limit = 50 µg/g.
[b]Average for one week.

TABLE 4-13. TRACE METALS RESULTS FOR COAL SAMPLES

Metal	Concentration, µg/gram				
	1	2	3	4	5[a]
Aluminum	5,080	3,740	5,070	4,720	4,470
Boron	22.2	21.3	21.8	27.7	23.8
Barium	56.3	47.6	53.2	51.4	61.9
Beryllium	0.8	0.7	0.7	0.7	0.7
Calcium	1,000	1,030	1,230	1,670	1,740
Cadmium	ND[b]	ND	ND	ND	ND
Cobalt	3.9	4.2	3.2	7.8	4.1
Chromium	13.7	12.9	11.0	11.8	12.9
Copper	12.9	9.8	11.0	20.7	16.1
Iron	11,800	9,580	6,060	29,000	7,930
Magnesium	328	318	390	562	452
Manganese	13.9	14.9	11.2	30.3	16.7
Molybdenum	ND	ND	ND	ND	ND
Sodium	3,310	2,860	2,860	3,680	2,660
Nickel	ND	ND	ND	9.9	ND
Lead	12.8	ND	ND	12.4	ND
Tin	ND	ND	ND	ND	ND
Titanium	364	306	319	307	284
Vanadium	29.7	28.1	30.6	18.1	20.7
Yttrium	3.4	3.0	3.6	5.0	4.3
Zinc	2.8	6.7	2.7	5.7	5.2

[a]Average of lab duplicates.

[b]ND - not detected or ≤100 x detection limits in last column of Table 4-11.

TABLE 4-14. CLAY FEED, AGGREGATE PRODUCT AND SCRUBBER INFLUENT CONDITIONS

Run number	Chlorine			Flow rate		
	Clay feed, µg/g	Aggregate product, µg/g	Scrubber influent, mg/L	Clay[b] feed, ton/hr	Aggregate product[b], ton/hr	Scrubber influent, gal/min
1	ND[a]	ND	56.8	13.5	10	143
2	ND	ND	101	13.5	10	143
3	ND	ND	54.8	13.5	10	143
4	ND	ND	58.4	13.5	10	143
5	ND	ND	86.3	13.5	10	143

[a]ND - not detected; detection limit = 50 µg/g.

[b]Values supplied by Florida Solite Corp.

not measured but assumed to be equal to the scrubber effluent flow rate. Clay feed and aggregate product rates are based on ton/hr rates provided by the Florida Solite Corporation that are measured approximately twice per year. Yearly production rates are based on 10 ton/hr at 70% operating hours per year.

Trace metals results for clay feed and aggregate product are shown in Table 4-15.

Trace metal results for the scrubber influent are shown in Table 4-16. Scrubber influent water shows relatively low concentraton for most of the trace metals. Only sodium, calcium, and magnesium have concentrations greater than 14 mg/L; all other metals are less than 1 mg/L.

4.3.4 Scrubber Effluent

Results from the analysis of scrubber effluent water for chlorine, hexavalent chromium, lead by atomic absorption, POHCs, flow rate and pH are shown in Table 4-17. Chlorine concentrations in the scrubber effluent range from 3 to 6 times those in the scrubber influent water shown earlier in Table 4-14. Hexavalent chromium was not detected in filtered samples. Although the samples were filtered, ample solid sample volume was not available to analyze Cr+6 in the solid phase. A sample from Run 3 was analyzed prior to filtering. The total Cr+6 was not detected (<10µg/L) showing that by subtraction, the Cr+6 concentration in the solids was also not detected (<10µg/L).

Lead (by atomic absorption) was found in both filtrate and solid portions of the effluent water in concentrations ranging from 6.75 mg/L to 20.97 mg/L and 6.92 mg/L to 40.64 mg/L, respectively. The scrubber effluent saples were particulate laden and ranged from 3% to 10% solids by weight.

TABLE 4-15. TRACE METALS RESULTS FOR CLAY FEED AND AGGREGATE PRODUCT

Metal	Clay feed concentration, µg/g					Aggregate product concentration, µg/g				
	1	2	3	4	5	1	2	3	4	5
Aluminum	26,100	27,400	30,200	34,000	27,700	14,600	7,950	12,100	3,980	8,550
Boron	7.9	9.5	12.7	11.9	9.7	7.0	13.0	4.2	4.6	5.3
Barium	37.8	38.0	35.7	48.4	34.9	20.6	11.4	23.5	9.8	16.9
Beryllium	1.2	1.3	1.9	1.8	1.4	0.7	0.4	0.6	0.2	0.4
Calcium	1,520	1,990	3,300	2,510	2,140	1,190	548	1,250	624	854
Cadmium	ND[b]	ND	ND	ND	ND	ND	ND	ND	ND	ND
Cobalt	6.4	7.4	15.5	8.0	7.3	8.0	4.9	6.3	6.8	7.3
Chromium	60.4	64.6	68.0	65.3	61.3	33.6	20.7	28.9	18.7	27.7
Copper	18.3	20.8	14.9	10.8	21.4	12.0	8.4	12.3	15.0	14.9
Iron	16,100	17,000	20,400	22,200	15,700	12,000	6,820	8,840	4,880	7,110
Magnesium	3,090	3,440	4,540	4,020	3,300	1,610	914	1,370	591	972
Manganese	40.5	51.0	72.3	53.8	54.3	56.7	27.5	30.8	18.7	28.2
Molybdenum	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Sodium	3,180	3,990	4,380	4,650	4,070	2,990	2,420	3,030	2,750	2,690
Nickel	15.6	13.7	26.2	15.5	20.1	9.2	ND	ND	7.6	8.8
Lead	27.8	35.5	34.7	31.0	34.6	22.3	13.2	15.2	ND	13.8
Tin	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Titanium	396	420	392	363	390	294	285	274	427	328
Vanadium	44.1	45.4	44.2	53.8	43.3	23.4	14.2	17.9	10.0	16.7
Yttrium	ND	ND	ND	ND	ND	5.1	3.8	6.6	3.5	5.1
Zinc	25.6	27.8	44.0	29.5	25.1	28.3	15.8	22.4	15.3	23.4

[a]Average of lab duplicates.

[b]ND - not detected for clay feed and aggregate product results (or ≤100 x detection limits in last column of Table 4-11).

TABLE 4-16. SCRUBBER INFLUENT TRACE METALS CONCENTRATIONS

Metal	Concentration in scrubber influent, mg/L					Detection limits, mg/L
	1	2	3	4	5	
Aluminum	0.22	0.22	0.34	0.17	0.42	0.06
Boron	0.06	ND[a]	0.45	0.55	ND	0.02
Barium	0.06	0.04	0.12	0.04	0.05	0.01
Beryllium	ND	ND	ND	ND	ND	0.002
Calcium	53.1	52.8	52.3	50.7	57.9	0.025
Cadmium	ND	ND	ND	ND	ND	0.012
Cobalt	ND	ND	ND	ND	ND	0.03
Chromium	ND	ND	ND	ND	ND	0.01
Chromium[b]	0.06[b]	0.04[b]	0.03[b]	0.09[b]	0.04[b]	0.03[b]
Copper	ND	ND	ND	ND	ND	0.02
Iron	0.45	0.40	0.44	0.39	0.5	0.045
Magnesium	14.5	14.8	14.9	14.7	16.9	0.03
Manganese	0.004	0.003	ND	ND	0.004	0.002
Molybdenum	ND	ND	ND	ND	ND	0.05
Sodium	27.8	27.8	27.4	25.3	37.2	6.0
Nickel	ND	ND	ND	ND	ND	0.065
Lead	ND	ND	ND	ND	ND	0.095
Lead[c]	0.21[c]	ND[c]	ND[c]	ND[c]	0.07[c]	0.043[c]
Tin	ND	ND	ND	ND	ND	0.550
Titanium	ND	ND	ND	ND	0.01	0.01
Vanadium	ND	ND	ND	ND	ND	0.03
Yttrium	ND	ND	ND	ND	ND	0.025
Zinc	0.05	ND	ND	ND	ND	0.015

[a] ND - not detected; detection limits shown in last column.

[b] Chromium samples analyzed by atomic absorption; units in μg/L.

[c] Lead samples analyzed by atomic absorption.

Only methyl ethyl ketone and toluene were found in concentrations greater than the detection limits in samples 2, 3, and 4. Methyl isobutyl ketone and tetrachloroethylene were not detected nor had values close to the detection limits.

Scrubber effluent pH also is shown in Table 4-17 and ranged from 2.4 to 4.3. The pH measurements were not made in the field but on samples returned to the MRC-Dayton Laboratory; however, results of measurements made in the field using pH paper showed all samples to be within the pH range of 2.4 to 4.3.

Trace metals results for filtered scrubber effluent water are shown in Table 4-18. Trace metals of highest concentrations in filtered effluent include aluminum and calcium (>75 mg/L), sodium and magnesium (18-60 mg/L), and iron, lead, and zinc (1-10 mg/L). All other trace metals analyzed by ICP were less than 1 mg/L. Analyses of solids in scrubber effluent water show aluminum and iron in highest concentration (12,000-25,000 µg/g), followed by sodium (9,000-17,000 µg/g), lead and titanium (2,000-6,000 µg/g), calcium and magnesium (1,000-3,000 µg/g), and barium and zinc (400-1,300 µg/g). All other ICP trace metals were less than 200 µg/g.

EP toxicity test results, shown in Table 4-19, indicate average extracted metal concentrations for Days 1-5 below RCRA levels, which are also listed in the table. Although the average lead concentration is less than the RCRA level, two samples exceed the 5,000 µg/L RCRA level. Due to the nature of the waste samples, only metals and not the organic/pesticide compounds in the extract were analyzed.

TABLE 4-17. SCRUBBER EFFLUENT WATER CONDITIONS

Run number	Chlorine, mg/L	Chromium^{+6} in filtrate[a], µg/L	Lead, mg/L		POHC, µg/L				Flow rate, gal/min	pH
			Filtrate	Solids	MEK	MIBK	Perc	Toluene		
1	253	ND[b]	6.75	40.64	ND[b]	ND[b]	ND[c]	0.43	143	3.4
2	368	ND[b]	20.97	17.24	250	13	ND[c]	280	143	2.4
3	307	ND[d]	7.15	38.56	210	16	ND[c]	310	143	2.6
4	205	ND[b]	7.37	6.92	180	24	ND[c]	270	143	4.3
5	140	ND[b]	8.17	11.22	9	12	ND[c]	0.87	143	4.1

[a]Filtered sample.

[b]ND - not detected; detection limit for Cr^{+6}, MEK, and MIBK = 5 µg/L.

[c]ND - not detected; detection limit for Perc = 1 µg/L.

[d]Total Cr^{+6} in sample is ND (<10 µg/L).

TABLE 4-18. SCRUBBER EFFLUENT TRACE METAL CONCENTRATIONS

Metal	Filtered scrubber effluent, mg/L					Scrubber effluent solids, µg/g				
	1	2	3	4	5	1	2	3	4	5
Aluminum	91.6	97.7	99.2	81.8	85.1	12,400	13,000	13,000	22,600	22,300
Boron	0.68	0.68	0.70	0.84	0.81	ND	ND	ND	9.8	ND
Barium	ND[a]	ND	ND	ND	ND	1,010	1,290	1,270	800	886
Beryllium	0.01	0.01	0.01	0.01	0.01	3.4	3.8	3.8	2.5	2.7
Calcium	85.6	76.9	78.9	112.0	87.3	1,800	1,530	1,800	2,710	1,840
Cadmium	0.15	0.21	0.21	0.30	0.10	ND	ND	ND	10.2	7.3
Cobalt	0.07	0.07	0.07	0.07	0.07	45.4	49.7	46.3	39.4	28.9
Chromium	0.23	0.23	0.24	0.21	0.12	159	194	194	411	189
Copper	0.58	0.64	0.65	0.34	0.41	107	132	133	150	118
Iron	7.01	9.47	10.2	1.78	3.29	23,600	25,200	25,100	21,200	19,800
Magnesium	19.4	18.3	19.6	21.7	19.4	1,780	1,850	1,830	2,660	2,000
Manganese	0.40	0.37	0.37	0.49	0.41	64.8	66	65.4	68.0	59.8
Molybdenum	0.11	0.17	0.18	ND	ND	ND	ND	ND	ND	ND
Sodium	52.8	54.2	56.6	51.1	50.7	14,700	17,000	17,300	13,300	9,360
Nickel	0.14	0.14	0.15	0.17	0.15	61.3	67.7	65.1	42.6	43.7
Lead	2.83	3.18	3.29	1.35	1.57	3,990	6,470	6,400	2,220	3,520
Tin	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Titanium	0.10	0.11	0.14	0.07	0.02	2,020	2,540	2,990	3,500	1,750
Vanadium	0.24	0.25	0.25	ND	ND	39.7	42.1	39.7	45.5	60.4
Yttrium	NA[b]	NA	NA	NA	NA	NA	NA	NA	NA	NA
Zinc	2.51	3.21	3.28	1.65	1.58	785	1,030	1,040	363	441

[a] ND - Not detected.

[b] NA - Not analyzed.

TABLE 4-19. EP TOXICITY RESULTS ON SCRUBBER DISCHARGE WATER, KILN NO. 5 FLORIDA SOLITE CORPORATION, FEBRUARY 21-25, 1983

Compound	Concentration, µg/L						
	Sample 1	Sample 2	Sample 3[a]	Sample 4[a]	Sample 5[a,b]	Average (Samples 1 through 5)	Level specified by RCRA
Arsenic	168	450	100	65	73	171	5,000
Selenium	30	38	<50	<50	<50	<44	1,000
Mercury	5.3	<1.0	<1.0	1.4	5.0	2.7	200
Silver	<10	<10	<10	<10	<10	<10	200
Barium	24,900	42,900	55,900	30,400	41,450	39,110	100,000
Cadmium	152	212	212	251	78	181	1,000
Chromium	143	135	205	160	40	137	5,000
Lead	2,775	5,111	2,722	2,444	5,583	3,727	5,000

[a]Sample required extraction.

[b]Average of duplicate sample

SECTION 5

ANALYSIS OF RESULTS

5.1 DESTRUCTION AND REMOVAL EFFICIENCIES

The complex combustion chemistry for organic materials becomes perplexing when a broad range of organic compounds present in a liquid waste are burned. On a weight basis, most of the organic carbon in the waste is oxidized to CO_2 in the combustion process, but trace amounts of organic chemicals survive the oxidation process and are only partially reacted or in some cases, new organic chemicals may be produced. These new surviving compounds are often referred to as products of incomplete combustion (PICs). Accordingly, the test burn investigated the amount of destruction of the organic compounds in the hazardous waste.

Destruction and removal efficiency (DRE) for an incineration/air pollution control system is defined by the following equation:

$$DRE = \frac{W_{in} - W_{out}}{W_{in}} (100) \qquad (5\text{-}1)$$

Where DRE = destruction and removal efficiency,%
W_{in} = Mass feed rate of principal organic hazardous constituent(s) (POHCs) fed to the incinerator
W_{out} = mass emission rate of principal organic hazardous constituent(s) (POHCs) to the atmosphere (as measured in stack prior to discharge).

DRE calculations are based on combined efficiencies of the destruction of the POHC in the incinerator, or the aggregate kiln, and the removal of the POHC from the gas stream in the air

pollution control system. The presence of POHCs in liquid/solid discharges from the air pollution control devices is not accounted for in the DRE calculation as currently defined by the EPA. RCRA Part 264, Subpart 0 regulations for hazardous waste incinerators require a DRE of 99.99% for all principal organic hazardous constituents of a waste unless it can be demonstrated that a higher or lower DRE is more appropriate based on human health criteria. Specification of the POHCs in a waste is subject to best engineering judgment, considering the toxicity, thermal stability, and quantity of each organic waste constituent. DRE requirements in the Subpart 0 regulations do not apply to metals or other non-combustible materials. Aggregate kilns are currently exempted from hazardous waste trial burns by federal regulations.

The POHCs chosen in this test program were four compounds known to be present in the current fuel mixture:

- Methyl ethyl ketone (MEK)
- Methyl isobutyl ketone (MIBK)
- Tetrachloroethylene (Perc)
- Toluene

Toluene, MEK, and Perc are RCRA Part 261, Appendix VIII compounds; MIBK is not listed. Toluene, MEK, and MIBK were present in high concentration for organic compounds (see Table 4-9). Perc was selected because the chlorinated hydrocarbons are, in general, difficult to thermally destroy and Perc was the only chlorinated organic measured in the top twenty constituents of the waste fuel.

These four POHCs were sampled in the exhaust gas by the volatile organic sampling train (VOST) and analyzed by gas chromatography/ mass spectrometry (GC/MS). Results of the analysis for exhaust gas concentration of POHCs (W_{out}) were presented in Table 4-7. Grab samples of waste fuel feed were composited for daily samples and analyzed by GC/MS. Results of the mass feed rate for each POHC (W_{in}) were presented in Table 4-10.

The DREs calculated using Equation 5-1 for each test run for POHCs are shown in Table 5-1. Methyl ethyl ketone (MEK) was destroyed and removed to at least 99.99% efficiency. Only two runs showed MEK DREs less than 99.99%: Runs $3A^3$ and $3B^3$. Runs $3A^3$ and $3B^3$ were side-by-side runs (with 3A and 3B) that were split with the EPA QA contractor. Runs $3A^3$ and $3B^3$ do not show good comparison with Runs 3A and 3B for MEK, possibly owing to high blank contamination problems on the QA contractor field blanks. Run $3B^3$ is an outlier and is not considered a significant part of the data. The overall DRE average for MEK for all five days was 99.998% ± 0.006% (95% confidence limits).

DREs for methyl isobutyl ketone (MIBK) ranged from 99.986% to $\geq$99.999%. The 99.986% value was the only MIBK DRE less than 99.992%. The overall average for MIBK was 99.998% ± 0.006% (95% confidence limits).

DREs for tetrachloroethylene (Perc) ranged from 99.993% to $\geq$99.999%. Excellent consistency was found for each day of sampling. Split samples on Day 3 (Runs $3A^3$, $3B^3$, and $3D^1$) all showed low relative difference. The overall DRE average for Perc was 99.997% ± 0.004% (95% confidence limits).

DREs toluene ranged from 99.995% to >99.999%. The overall average for toluene was 99.999% ± 0.002% (95% confidence limits), making toluene the easiest POHC to destroy and remove.

Based on Table 4-7, W_{out} values for Run 2I for all POHCs, Runs 2J, 2K, and 5C for MEK, and Run 2F for Perc and toluene are essentially zero, because front tubes are <2 times blank tubes, which is within the sensitivity of the GC/MS instrument. By definition (Equation 5-1), the DRE = 100.0% for W_{out} = 0; however, because analysis of tubes is within the sensitivity of the GC instrument, a conservative approach was taken and the DRE was presented as >99.999% for the above mentioned runs.

During sampling, approximately 5 mL of moisture was condensed in the VOST impinger prior to the Tenax tubes. The chance that POHCs were contained in the condensate is high, however, since the condensate was not saved, exact POHC concentration was not determined. In addition, if POHCs were in the condensate, they were not accounted for in the calculation of W_{out} or DRE. The effect the POHCs in the condensate has on DREs was estimated by assuming 1) 5 mL of condensate was collected in each run; and 2) the concentration of POHCs in the condensate equals the scrubber effluent concentration. Multiplying the scrubber effluent POHC concentrations in Table 4-17 by 5 mL yields the estimated mass (ng) of each POHC in the VOST condensate for each run as shown in Table 5-2. Mass values in Table 5-2 are added to the mass values on the Tenax tubes (Table 4-6), and new POHC stack emission values (W_{out}) and DREs are calculated. The results of these calculations, shown in Table 5-2 have only a minor effect on DREs. Methyl ethyl ketone results were reduced by an average of 0.005% (DRE reduction from 99.998% to 99.993%). Methyl isobutyl ketone results were reduced by only 0.001% from a 99.998% DRE to 99.997%. Tetrachloroethylene was not reduced at all since POHC concentrations in the scrubber effluent were not detected. Toluene results were reduced by 0.002% from a 99.999% DRE to 99.997%.

5.2 CHLORINE MATERIAL BALANCE

The combustion of chlorinated hazardous wastes as fuel in aggregate kilns results in the generation of hydrogen chloride (HCl) in the kiln. Hydrogen chloride can be absorbed by the aggregate product or aggregate kiln lining, removed by the scrubber, or emitted as part of the particulate. Nonabsorbed HCl will be emitted from the stack as will the unburned chlorinated hydrocarbons. The chlorine material balance will be presented to demonstrate the fate of chlorine in the kiln process.

TABLE 5-1. DESTRUCTION AND REMOVAL EFFICIENCIES OF POHCs

Run number	DRE, %[a] POHC 1 (MEK)	POHC 2 (MIBK)	POHC 3 (Perc)	POHC 4 (Toluene)
2F	99.999	99.999	>99.999	>99.999
2G	99.999	99.999	99.999	99.999
2H	99.999	99.999	99.999	99.999
2I	>99.999	>99.999	>99.999	>99.999
2J	>99.999	99.999	99.999	99.999
2K	>99.999	99.999	99.999	99.999
Day 2 Average	99.999	99.999	99.999	99.999
3A	99.999	99.998	99.998	99.999
3A[3]	99.985	99.999	99.998	99.999
3B	99.999	99.999	99.999	99.999
3B[3]	99.968	99.998	99.999	99.999
3D	99.999	99.999	99.999	99.999
3D[1]	99.999	99.999	99.999	99.999
Day 3 Average	99.992	99.999	99.999	99.999
4A	99.998	99.986	99.993	99.995
4B	99.999	99.992	99.995	99.997
4C	99.999	99.998	99.997	99.998
4D	99.999	99.996	99.997	99.998
4E	99.999	99.997	99.998	99.998
4F	99.999	99.998	99.998	99.999
4G	99.999	99.994	99.997	99.999
4H	99.999	99.997	99.998	99.998
Day 4 Average	99.999	99.995	99.997	99.998
5A	99.999	99.999	99.995	99.999
5B	99.999	99.999	99.997	99.999
5C	>99.999	99.999	99.995	99.999
5E	99.999	99.999	99.991	99.998
5F	99.999	99.999	99.995	99.999
Day 5 Average	99.999	99.999	99.995	99.999
Overall Average	99.998	99.998	99.997	99.999

[a]Run 1A, 2A through 2E, and 5D were voided in the field or during analysis.

Figure 5-1 shows the schematic diagram for the aggregate kiln. The chlorine material balance used the following process streams:

INPUTS	OUTPUTS
Coal	Aggregate product
Waste fuel	Scrubber effluent water
Scrubber influent water	Stack gas
Clay feed	

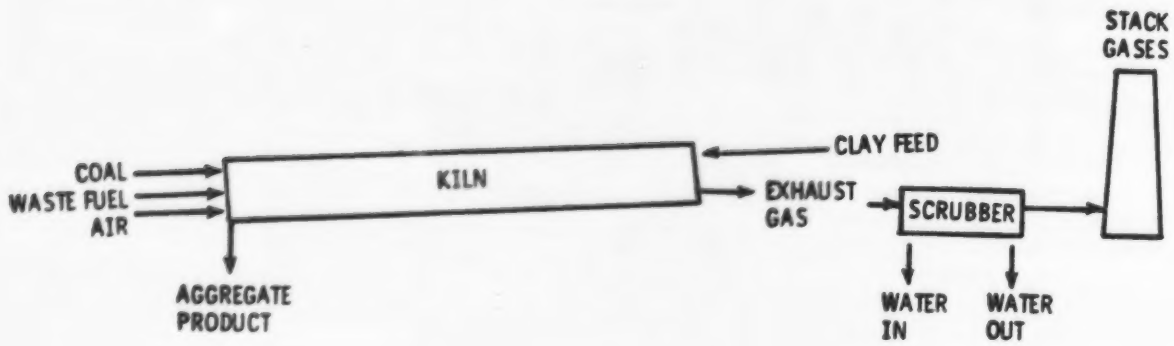

Figure 5-1. Schematic diagram of aggregate kiln process.

Table 5-2 shows the results of the chlorine material balance tests in the aggregate kiln. The material balance closure for chlorine ranged from 63.9% to 116.5% and averaged 82.1% (100% closure is ideal). The coal, clay feed (inputs), and aggregate product (output) were assumed to be zero. If coal, clay, and aggregate values are included and maximum rates assumed, the chlorine balance values are included and maximum rates assumed, the chlorine balance closure result improves by approximately 1% for each run. Possible explanations for closures not being 100% include (1) chlorine absorbed by the kiln walls, (2) chlorine in the particulate, (3) chlorine in the fugitive emissions at the burner end and exhaust

TABLE 5-2. EFFECT OF POHCs IN VOST CONDENSATE ON DRE

Run number	POHCs in VOST Condensate[a], ng				Revised DRE (change from Table 5-2 DRE), %			
	MEK	MIBK	Perc	Toluene	MEK	MIBK	Perc	Toluene
1	ND[b]	ND[b]	ND[b]	2.2	-[d]	-	-	-
2	1,250	65	ND[b]	1,400	99.993[d] (.006)	99.999 (0)	99.999 (0)	99.998 (.001)
3	1,050	80	ND[b]	1,550	99.990 (.008)	99.998 (.001)	99.999 (0)	99.996 (.003)
4	900	120	ND[b]	1,350	99.994 (.005)	99.993 (.002)	99.997 (0)	99.995 (.003)
5	45	60	ND[b]	4.4	99.999 (0)	99.999 (0)	99.995 (0)	99.999 (0)

[a]Assume 5 mL of condensate per run, and POHC concentration in VOST condensate equals POHC concentration in scrubber effluent.

[b]Not detected.

[c]No runs made.

[d]Runs 3A[3] and 3B[3] not included due to possible contamination problems.

TABLE 5-3. CHLORINE MATERIAL BALANCE

Run number	Materials to kiln, lb/hr					Materials from kiln, lb/hr				Material balance closure, %
	Coal	Waste fuel	Scrubber influent	Clay feed	Total In[a]	Aggregate product	Scrubber effluent	Stack gas[c]	total out[b]	
1	<0.08	23.8	4.1	<1.4	27.9	<1.0	18.1	0.05	18.2	65.2
2	<0.08	31.3	7.2	<1.4	38.5	<1.0	26.3	0.05[d]	26.4	68.6
3	<0.08	19.0	3.9	<1.4	22.9	<1.0	22.0	0.02	22.0	96.1
4	<0.08	8.5	4.2	<1.4	12.7	□1.0	14.7	0.08	14.8	116.5
5	<0.08	9.6	6.2	<1.4	15.8	[1]1.0	10.0	0.06	10.1	63.9

[a]Assume coal and clay feed are zero and not included in total in value.

[b]Assume aggregate product is zero and not included in total out value.

[c]Values for unburned chlorinated organics sampled by VOST not included because all runs were □ 7 x 10^{-5} lb/hr stack gas emission.

[d]No run made; use Run No. 1 value.

gas emitted from the scrubber discharge pipe, (4) random error associated with chlorine sampling and analysis results, or (5) errors in estimated rates for scrubber influent and effluent streams.

5.3 TRACE METALS MATERIAL BALANCE

The trace metals fed to the kiln by the coal, waste fuel, scrubber influent water, and clay feed input materials can be adsorbed by the aggregate product or kiln lining, removed by the scrubber, or emitted as part of the particulate. Nonabsorbed metals will be emitted from the stack. A trace metals materials balance will be presented to demonstrate the fate of the metals in the kiln process.

Table 5-3 summarizes metal material balance results for all five runs, showing the percent entering or exiting the kiln process by each stream. Typically, the day feed contributes 60% to 100% and the waste fuel contributes 1% to 30% of a metal to the kiln process. The coal and scrubber influent water contribute negligible amounts. However, cadmium, molybdenum, lead, and zinc are primarily in the waste fuel (70% to 100%).

The aggregate product and solids in the scrubber effluent typically contain the majority of metals exiting the kiln. Usually the stack gas and scrubber effluent liquid phase contain a low percentage of metals exiting the kiln.

Tables 5-4 through 5-8 show the results of the trace metal balance for each run. The range of material balance closure for all metals and all runs is 16% to 521% and the average is 99% ± 130% (95% confidence limits). The material balance was calculated by dividing the total mass out by total mass in. Although the average closure is near 100%, the range is extremely wide, as indicated by the high standard deviation of 130%. Causes for the balance being less than 100% include the nonmeasurement of metals

TABLE 5-4. AVERAGE CONTRIBUTION OF METALS BY PROCESS STREAMS

Metal	Percent to kiln				Percent from kiln				
	Coal	Waste fuel	Scrubber influent	Clay feed	Aggregate product	Scrubber Effluent Liquid	Scrubber Effluent Solid	Stack gas	Average closure%
Aluminum	1	<1	<1	98	66	2	32	<1	35
Boron	8	15	5	72	64	26	9	<1	53
Barium	4	43	<1	53	8	<1	92	<1	254
Beryllium	3	<1	<1	97	41	3	56	<1	53
Calcium	2	27	4	67	54	18	28	<1	34
Cadmium	<1	100	<1	<1	<1	53	33	14	153
Cobalt	2	28	<1	70	44	2	54	<1	100
Chromium	1	28	<1	71	36	1	62	1	79
Copper	3	37	<1	60	31	5	63	1	122
Iron	4	1	<1	95	61	<1	39	<1	45
Magnesium	1	1	1	97	65	4	31	<1	28
Manganese	2	11	<1	87	65	3	23	<1	61
Molybdenum	<1	100	<1	<1	<1	43	<1	57	23
Sodium	4	11	1	84	48	3	49	<1	96
Nickel	<1	7	<1	93	22	4	70	4	71
Lead	<1	85	<1	15	2	1	96	3	318
Tin	ND[a]	ND[a]	ND[a]	ND[a]	ND[a]	ND[a]	ND[a]	ND[a]	ND[a]
Titanium	4	2	<1	94	40	<1	60	<1	180
Vanadium	3	<1	<1	97	60	3	37	<1	38
Yttrium	97	<1	3	<1	100	<1	<1	<1	1,300
Zinc	<1	68	<1	32	13	5	80	2	45

[a]Not detected

TABLE 5-5. TRACE METALS MATERIAL BALANCE FOR RUN 1

Metal	Materials in, g/hr					Materials out, g/hr					Material balance closure, %
	Coal	Waste fuel	Scrubber influent	Clay feed	Total in	Aggregate product	Scrubber effluent, filtered	Scrubber effluent, solids	Stack gas particulate	Total out	
Aluminum	3,556	1,911	7.1	319,933	325,407	132,560	2,864	14,880	168	150,480	46
Boron	15.5	26.5	1.9	96.84	140.7	63.6	21.3	ND	0.9	85.9	61
Barium	39.4	349.2	1.9	463.4	853.9	187.0	ND	1,212	3.2	1,600	188
Beryllium	0.56	ND[a]	ND	14.7	15.3	6.4	0.3	4.1	0.03	10.8	71
Calcium	700	17,127	1,724	18,632	38,183	10,805	2,676	2,160	97.7	15,739	41
Cadmium	ND	17.1	ND	ND	17.1	ND	4.7	ND	2.2	5.9	35
Cobalt	2.7	38.8	ND	78.5	120	72.6	2.2	54.5	0.4	129	108
Chromium	9.6	38.1	ND	740.4	788.1	305.1	7.2	191	5.5	509	64
Copper	9.0	101.4	ND	224.3	344.7	108.9	18.1	128	5.3	260	76
Iron	8,260	1,995	14.6	197,354	207,625	108,960	219	28,320	139	137,638	66
Magnesium	229	804.5	470.8	37,877	39,382	14,619	607	2,136	42.5	17,404	44
Manganese	9.7	93.9	0.1	496.4	600	514.8	12.5	77.8	1.5	607	101
Molybdenum	ND	21.4	ND	ND	21.4	ND	3.4	ND	3.5	6.9	32
Sodium	2,317	6,883	902.8	38,980	49,083	27,150	1,651	17,640	230	46,671	95
Nickel	ND	17.1	ND	191.2	208	83.5	4.4	73.6	5.1	166	80
Lead	9.0	1,973	ND	340.8	2,323	202.5	88.5	4,788	142	5,321	229
Tin	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Titanium	255	161.9	ND	4,854	5,270	2,669	3.1	2,424	6.2	5,102	97
Vanadium	20.8	ND	ND	540.8	562	212.7	7.5	47.6	0.7	269	48
Yttrium	2.4	ND	ND	ND	ND	46.3	NA[b]	NA	0.2	ND	ND
Zinc	2.0	1,022	1.6	313.8	1,339	257.0	78.5	942	28.9	1,307	98

[a] ND - not detected.

[b] NA - not analyzed.

TABLE 5-6. TRACE METALS MATERIAL BALANCE FOR RUN 2

Metal	Materials in, g/hr					Materials out, g/hr					Material balance closure, %
	Coal	Waste fuel	Scrubber influent	Clay feed	Total in	Aggregate product	Scrubber effluent, filtered	Scrubber effluent, solids	Stack gas particulate[b]	Total out	
Aluminum	2,618	1,885	7.2	335,869	340,379	72,186	3,074	13,000		88,260	26
Boron	14.9	29.0	ND	116	159.9	118	21.4	ND		139.4	87
Barium	33.3	350	1.3	466	850.6	104	ND	1,290		1,394	164
Beryllium	0.5	ND	ND	16	16.5	3.6	0.3	3.8		7.7	47
Calcium	721	14,370	1,715	24,993	41,799	4,976	2,419	1,530		8,925	21
Cadmium	ND	17.5	ND	ND	17.5	ND	6.6	ND		6.6	38
Cobalt	2.9	39.0	ND	90.7	132.6	44.5	2.2	49.7		96.4	73
Chromium	9.0	416	ND	792	1,217	188	7.2	194		389	32
Copper	6.7	108	ND	255	370	76.3	20.1	132		228	62
Iron	6,706	1,995	13.0	208,386	217,100	61,926	297.6	25,200		87,424	40
Magnesium	223	687	480	42,167	43,557	8,299	575.8	1,850		10,725	25
Manganese	10.4	92.9	0.1	625	728	250	11.6	66.0		328	45
Molybdenum	ND	28.8	ND	ND	28.8	ND	5.3	ND		5.3	18
Sodium	2,002	7,460	903	48,909	59,274	21,973	1,705	17,000		40,678	69
Nickel	ND	17.0	ND	168	185	ND	4.4	67.7		72.1	39
Lead	ND	2,218	ND	435	2,653	120	100	6,470		7,311	276
Tin	ND	ND	ND	ND	ND	ND	ND	ND		ND	ND
Titanium	214	143	ND	5,148	5,505	2,587	3.5	2,540		5,130	93
Vanadium	19.7	ND	ND	557	577	129	7[c]	42.1		179	31
Yttrium	2.1	ND	0.3	ND	2.4	34.5	NA[c]	NA		34.5	NA
Zinc	4.7	1,052	ND	341	1,398	143	101	1,030		1,274	91

[a] ND - not detected.

[b] No EPA 5 run made on Day 2.

[c] NA - Not analyzed.

TABLE 5-7. TRACE METALS MATERIAL BALANCE FOR RUN 3

Metal	Materials in, g/hr					Materials out, g/hr					Material balance closure, %
	Coal	Waste fuel	Scrubber influent	Clay feed	Total in	Aggregate product	Scrubber effluent, filtered	Scrubber effluent, solids	Stack gas particulate	Total out	
Aluminum	3,549	1,549	11.0	370,192	375,301	109,868	3,122	12,870	244	126,104	34
Boron	15.3	23.5	14.6	157	210	38.1	22.0	ND	1.0	61.1	29
Barium	37.2	285	3.9	438	764	214	ND	1,257	5.3	1,476	193
Beryllium	0.5	ND[a]	ND	23	23.5	5.4	0.3	3.8	0.0	9.5	40
Calcium	861	10,555	1,698	40,451	53,565	11,350	2,483	11,782	149	15,764	29
Cadmium	ND	16.0	ND	ND	16.0	ND	6.6	ND	3.3	9.9	62
Cobalt	2.2	41.0	ND	190	233	57.2	2.2	45.8	0.4	106	45
Chromium	7.7	374	ND	833	1,215	262	7.6	192	9.1	471	39
Copper	7.7	130	ND	183	321	112	20.5	132	7.4	272	85
Iron	4,242	1,869	14.3	250,063	256,188	80,267	321	24,849	211	105,648	41
Magnesium	273	558	484	55,651	56,966	12,439	617	1,812	59.3	14,927	26
Manganese	7.8	80.5	ND	886	974	280	11.6	65	2.4	359	37
Molybdenum	ND	22.8	ND	ND	22.8	ND	5.7	ND	3.1	8.8	39
Sodium	2,002	7,960	890	53,690	64,542	27,512	1,782	17,127	257	46,678	72
Nickel	ND	15.3	ND	321	336	ND	4.7	64.5	4.7	73.9	22
Lead	ND	2,043	ND	426	2,469	138	104	6,336	191	6,769	274
Tin	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Titanium	223	129.5	ND	4,805	5,157	2,488	4.4	2,960	11.2	5,463	106
Vanadium	21.4	ND	ND	542	563	163	7[b]	39.3	1.2	211	38
Yttrium	2.5	ND	ND	ND	2.5	59.9	NA[b]	NA	0.2	60.1	240
Zinc	1.9	937	ND	539	1,478	203	103	1,030	41.2	1,377	93

[a]ND - not detected.

[b]NA - Not analyzed.

TABLE 5-8. TRACE METALS MATERIAL BALANCE FOR RUN 4

Metal	Materials in, g/hr					Materials out, g/hr					Material balance closure, %
	Coal	Waste fuel	Scrubber influent	Clay feed	Total in	Aggregate product	Scrubber effluent, filtered	Scrubber effluent, solids	Stack gas particulate	Total out	
Aluminum	3,304	1,508	5.5	416,772	421,589	36,138	2,229	117,068	284	155,719	37
Boron	19.4	22.6	17.9	146	206	41.8	22.9	50.8	1.9	117	57
Barium	36.0	514	1.3	593	1,144	89.0	ND	4,144	6.4	4,239	371
Beryllium	0.5	ND[a]	nd	22.0	22.5	1.8	0.3	13.0	0.1	15.2	69
Calcium	1,169	7,287	1,647	30,767	40,870	5,666	3,052	14,038	194	22,950	56
Cadmium	ND	17.5	ND	ND	17.5	ND	8.2	52.8	3.6	64.6	369
Cobalt	5.5	51.8	ND	98.0	155	61.7	1.9	204	0.7	268	173
Chromium	8.3	252	ND	800	1,060	170	5.7	2,129	8.9	2,313	218
Copper	14.3	180	ND	132	327	136	9.3	777	7.9	930	285
Iron	20,300	3,179	12.7	272,128	295,620	44,310	48.5	109,816	229	154,404	52
Magnesium	393	661	477	49,277	50,808	5,366	591	13,778	72.6	19,808	39
Manganese	21.2	95.2	ND	659	775	170	13.4	352	3.1	539	69
Molybdenum	ND	9.7	ND	ND	9.7	ND	ND	ND	3.5	3.5	36
Sodium	2,576	4,444	821	57,000	64,841	24,970	1,393	68,894	305	95,562	147
Nickel	6.9	16.8	ND	190	214	69.0	4.6	220	8.5	302	141
Lead	8.7	1,870	ND	380	2,258	ND	36.7	11,499	221	11,757	521
Tin	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Titanium	215	88.2	ND	4,449	4,752	3,877	1.9	18,130	12.6	22,022	463
Vanadium	13.1	ND	ND	659	672	90.8	ND[b]	236	1.2	328	49
Yttrium	3.5	ND	ND	ND	3.5	31.8	NA[b]	NA	0.3	32.1	NA
Zinc	4.0	492	ND	361	857	139	45.0	1,880	48.0	2,112	246

[a]ND - not detected.

[b]NA - not analyzed.

TABLE 5-9. TRACE METALS MATERIAL BALANCE FOR RUN 5

Metal	Materials in, g/hr					Materials out, g/hr					Material balance closure, %
	Coal	Waste fuel	Scrubber influent	Clay feed	Total in	Aggregate product	Scrubber effluent, filtered	Scrubber effluent, solids	Stack gas particulate	Total out	
Aluminum	3,129	2,309	13.6	339,546	344,997	77,634	2,486	72,029	187	152,336	44
Boron	16.7	28.5	ND[a]	119	164	48.1	23.7	ND	1.7	73.5	45
Barium	43.3	530	1.6	428	1,003	153	ND	2,862	6.8	3,022	301
Beryllium	0.5	ND	ND	17.2	17.7	3.6	0.3	8.7	0.0	12.6	71
Calcium	1,218	6,083	1,880	26,232	35,413	7,754	2,550	5,943	105	16,352	46
Cadmium	ND	8.0	ND	ND	8.0	ND	2.9	23.6	1.5	28	350
Cobalt	2.9	33.5	ND	89.5	126	66.3	2.0	93.4	0.4	162	129
Chromium	9.0	690	ND	751	1,450	252	3.5	610	5.6	871	60
Copper	11.3	141	ND	262	414	135	12.0	381	6.3	534	129
Iron	5,551	2,547	16.2	192,451	200,565	64,559	96.1	63,954	162	128,771	64
Magnesium	316	50.7	549	40,451	41,367	8,826	567	6,460	43.9	15,897	38
Manganese	11.7	45.2	ND	665	722	256.0	12.0	193	1.5	463	64
Molybdenum	ND	46.2	ND	ND	46.2	ND	ND	ND	7.3	7.3	16
Sodium	1,862	5,893	1,208	49,890	58,853	24,425	1,481	30,324	163	56,393	96
Nickel	ND	15.9	ND	246	262	79.9	4.4	141	2.7	228	87
Lead	ND	3,134	ND	424	3,558	125.3	45.9	11,370	234	11,775	331
Tin	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Titanium	199	74.9	0.3	4,781	5,055	2,978	0.6	5,652	97	8,728	173
Vanadium	14.5	ND	ND	531	546	152	ND[b]	195	0.8	348	64
Yttrium	3.0	ND	ND	ND	3.0	46.3	NA[b]	NA	0.2	46.5	155
Zinc	3.6	677	ND	308	989	213	46.2	1,424	21.2	1,704	172

[a] ND - not detected.

[b] NA - not analyzed.

emitted as nonparticulates from the stack, metals emitted from gas with scrubber effluent water, metals that are absorbed by the kiln refractory, errors in flow rate measurements for scrubber influent and effluent waters, clay feed, and aggregate product that are approximately ±10%, and random errors in sampling and analyses of samples by ICAP.

SECTION 6

CONCLUSIONS

The purpose of this program was to: (1) conduct sampling and analysis during the burning of liquid hazardous organic waste as supplemental fuel; (2) calculate the DRE for each POHC; (3) determine the concentrations of particulate matter, SO_2, NO_x, and HCl in the stack gas; and (4) determine the fate of chlorine and trace metals in the stack gas and process samples by material balance. All of these objectives were met as described below:

- Consistent achievement of at least 99.99% destruction and removal efficiency was demonstrated for each POHC (MEK, MIBK, Perc, and toluene) during each test day. DREs for each POHC ranged and averaged as follows:

POHC	Range, %	Average, %
MEK	99.985 to >99.999	99.998
MIBK	99.986 to ≥99.999	99.998
Perc	99.991 to ≥99.999	99.997
Toluene	99.995 to ≥99.999	99.999

If POHCs in the condensate collected by the VOST train are included in the DRE calculation only the MEK DREs are reduced by (0.006%).

- Emissions of pollutants in the stack gas were determined and ranged as follows:

Pollutant	Range	
	kg/hr	lb/hr
Particulates	4.4 to 6.5	9.7 to 14.4
HCl	0.008 to 0.034	0.017 to 0.076
SO_2	72.2 to 99.6	159.1 to 219.4
NO_x	1.9 to 11.7	4.1 to 25.8

- Approximately 90% to 100% of the chlorine is fed to the kiln from the waste fuel and scrubber influent water. The fate of the chlorine is approximately 90% to 100% in the scrubber effluent water.

- Trace metals are fed to the kiln primarily by the clay feed (60% to 100%) and by the waste fuel (1% to 30%). The coal and limestone feed typically contribute negligible amounts of metals. The aggregate product and solids in the scrubber effluent typically contain the majority of metals exiting the kiln process (30% to 80% each). The stack gas and liquid phase of the scrubber effluent typically contain a low percentage of the metals exiting the kiln.

- The more toxic metals, such as lead, cadmium, and chromium, were fed to the kiln either in the waste fuel (cadmium and lead) or the clay feed (chromium). Almost all the lead exited the kiln in the solids in the scrubber effluent, while the chromium was distributed between the scrubber effluent solids and aggregate product and the cadmium was distributed in the scrubber effluent solid and liquid portions.

SECTION 7

QA/QC RESULTS

Quality assurance/quality control goals for precision and accuracy were set prior to initiating the project for the sampling and analyses conducted at the Florida Solite Corporation as shown in Tables 7-1 and 7-2 [see also Reference 1 in Section 3.3]. The project QA plan was reviewed and commented upon by IERL's-Cincinnati QA contractor, and in addition a QA systems audit was conducted by the contractor in the field. The following sections describe the QA/QC results as compared to the program goals.

7.1 STACK GAS SAMPLING AND ANALYSIS QA/QC RESULTS

Stack gas samples were taken and analyzed for particulates, HCl, metals, carbon dioxide, oxygen, sulfur dioxide, nitrogen oxides, and POHCs. Table 7-3 summarizes QA/QC results for all stack gas samples, except metals and POHCs, which will be presented later in this section. Sampling and analyses completeness goals established in Table 7-1 were not met, except for HCl as shown in Table 7-3. Particulate, SO_2, NO_x, and CO_2/O_2 sampling did not meet completeness goals owing to problems with the VOST train that delayed sampling or the plant shutdown on the morning of Day 3. All other QA/QC goals except one were met including the number of blanks taken, the number of splits performed and the percent relative difference, the number of standards analyzed and the percent recovery. One SO_2 spike was scheduled to be analyzed but the analysis was not performed.

TABLE 7-1. QA OBJECTIVES FOR PRECISION FOR EACH MAJOR MEASUREMENT

	Measurement parameter (method)	Number of samples	Number of blanks	Number of splits	Precision goals, relative difference, %		S&A completeness goals, %
					Within laboratory	Between laboratories	
1	Principal organic hazardous constituents (VOST,GC/MS)	31 pairs of sorbent tubes	10[a]	1[b]	25[b]	NA	95
2	Particulate matter (EPA 5)	5	2	NA	NA	NA	100
3	Hydrogen chloride (absorption, SIE)	5	1	1	10	15	100
4	Carbon dioxide and oxygen (EPA 3)	15	NA	5[c]	7.5 for CO_2 2.0 for O_2	NA	100
5	Nitrogen oxides (EPA 7)	40	2	4	10	15	95
6	Sulfur dioxide (EPA 6)	10	1	1	10	15	90
7	Waste fuel (grab → composite)	5	1	5[d]	10	15	100

(continued)

TABLE 7-1 (continued)

	Measurement parameter (method)	Number of samples	Number of blanks	Number of splits	Precision goals, relative difference, %		S&A completeness goals, %
					Within laboratory	Between laboratories	
8	Metals in particulate matter, waste fuel, coal, product, feed, scrubber influent and effluent (ICP)	40	8	8	15	NA	100
9	Chromium and lead in scrubber discharge (AAS)	10[e]	2	2	10	15	100
10	Hexavalent chromium in scrubber discharge (APHA 312B)	10[e]	2	2	10	NA	100

[a] Two field blank pairs (4), two trip blanks (2), two lab blanks (2), and two baseline lab blanks (2).

[b] This will be the only "sampling and analytical" split run in the program; a simultaneous side-by-side sample run will be conducted. All other splits refer to "analytical" splits only.

[c] EPA Method 3 is completed in triplicate until the dry molecular weights of any three analyses differ by no more than 0.3 g/g-mole of the mean value.

[d] Every waste fuel sample will be analyzed in duplicate.

[e] Each of the five scrubber discharge samples will be separated into sludge and supernatant fractions and analyzed separately.

NA = Not applicable.

TABLE 7-2. QA OBJECTIVES FOR ACCURACY FOR EACH MAJOR MEASUREMENT

	Measurement parameter (method)	No. of samples	No. of spikes	Reference standards	Accuracy goals, percent recovery	
					Spikes	Reference standards
1	Principal organic hazardous constituents (VOST, GC/MS)	31 pairs of sorbent tubes	32 to 62[a]	4 x 3[b]	NA	-[c]
2	Particulate matter (EPA 5)	5	NA	-[d]	NA	±5 of calibration factor for dry gas meter, ±0.2 for analytical balance
3	Hydrogen chloride (absorption, SIE)	5	1	5[e]	NA	NA[e]
4	CO_2 and O_2 (EPA 3)	15	NA	4[f]	NA	±5
5	Nitrogen oxides (EPA 7)	40	NA[g]	4	NA	±10
6	Sulfur dioxide (EPA 6)	10	1	1	±10	±10
7	Waste fuel (grab → composite)	5	NA	-[h]	NA	-[c]
8	Metals in various matrices, see Table 4-1 (ICP)	40	8 x 21[i]	8 x 21[i]	±20 (80-120%)	±10

(continued)

TABLE 7-2 (continued)

	Measurement parameter (method)	No. of samples	No. of spikes	Reference standards	Accuracy goals, percent recovery	
					Spikes	Reference standards
9	Chromium and lead in scrubber discharge (AAS)	10	2	2	±15	±10
10	Hexavalent chromium in scrubber discharge (APHA 312B)	10	2	2	±15	±10

[a]Each sorbent tube is spiked with an internal standard. The first analyses will be conducted on the front tube and back-up tube. If the gas volumes samples are sufficiently small such that break-through of the front tube does not occur, then the remaining back-up tubes will not be analyzed.

[b]Refers to each of four POHC external standards run at three levels bracketing the amount captured for sorbent tube.

[c]Rather than reference standards, external standards of each POHC are used for quantitation.

[d]Reference standards for particulate matter do not exist. EPA Method 5 is considered "accurate" if the dry gas meter, stack and meter temperature sensors, probe nozzle, and analytical balance demonstrate acceptable calibration.

[e]A five-point standards calibration curve is established for analysis.

[f]Bottled gases traceable to NBS containing 2-4% O_2, 15% O_2, 2-4% CO_2, and 14-18% CO_2.

[g]Not applicable because the whole sample is consumed during analysis.

[h]Each constituent >1% in the waste fuel is quantified by a separate external standard of the same chemical structure. All other constituents <1% in the waste are semi-quantified by comparison with an external standard which elutes in the vicinity of that constituent.

[i]Twenty-one different elements are spiked and have reference standards run.

TABLE 7-3. QA/QC RESULTS FOR STACK GAS SAMPLES

Parameter and units	Split samples: Result 1	Split samples: Result 2	Split samples: Average	Split samples: Relative diff., %	Standard samples: Observed	Standard samples: Actual	Standard samples: Recovery, %	Spiked samples: Sample value	Spiked samples: Spike concentration	Spiked samples: Observed value	Spiked samples: Recovery, %	Completeness goals, % (No. of actual samples/goals x 100)	No. of blanks (field and lab)
Particulate		NA				NA[a]			NA			80 (4/5)	3
HCl, μg	739	763	751	3.2		NA[b]			NA			100 (5/5)	2
SO_2[c], mL	394	397	396	0.8	396	381.3	104		NP[d]			80 (8/10)	2
NO_x, μg	156	163	160	4.4	160	159	101						
	155	159	157	2.6	157	159	98.7		NA[e]			88 (35/40)	4
	987	987	987	0	987	956	103						
	974	974	974	0									
NO_x, absorbance	0.23	0.22	0.23	4.4	974	956	102						
	0.29	0.29	0.29	0									
	0.26	0.27	0.27	3.7									
	0.27	0.28	0.28	3.6									
CO_2 and O_2[f], molecular wt.	29.59	29.68,29.68	29.58	0.3		NA[g]			NA			80 (12/15)	
	29.54	29.64,29.73	29.64	0.6									
	29.92	29.71,29.64	29.76	0.9									
	29.60	29.70,29.70	29.67	0.3									

[a]Not applicable; QA/QC for EPA method 5 includes calibration of sampling equipment and field blanks.

[b]Not applicable; five-point calibration curve is established for analysis.

[c]Each SO_2 titration is performed in duplicate.

[d]Not performed, although scheduled in Table 7-2.

[e]Not applicable because the whole sample is consumed during analysis.

[f]CO_2 and O_2 analyzed in triplicate.

[g]Bottled gases used to calibrate Fyrite prior to field use.

7.2 POHC STACK GAS SAMPLING AND ANALYSIS QA/QC RESULTS

Quantification of the POHCs in the stack gas was achieved by VOST sampling using Tenax tube adsorbent and thermal desorption GC/MS analyses of the Tenax tubes. Of the QA/QC goals specified in Table 7-1 and 7-2 for POHC stack gas analysis, all goals were met except the completeness goal and several of the split results. Table 7-4 summarizes QA/QC results for POHC sampling and analysis.

Thirty pairs of VOST samples were collected and returned to the MRC Dayton Laboratory; however, only twenty-five were analyzed because five runs were lost during analyses due to plugging or breakage (see Section 4.2.6). This reduced the sampling and analysis completeness from 97% to 81%, less than the 95% completeness goal.

Only one "within lab" split was specified; however, while in the field, two additional "between lab" split runs were made (side-by-side VOST systems) and samples were split with the EPA QA contractor. The "within lab" split at MRC shows excellant duplication and precision for all POHCs except MEK (relative difference = 152%). However, even a difference of 152% for MEK on Runs 3D and 3D[1] did not affect the final destruction efficiency of 99.999% for each run (see Table 5-1). VOST samples split between labs (MRC and QA contractor) showed good duplication for all POHCs except MEK. In this case, the DREs for MEK were affected (i.e., Run 3A DRE = 99.999%, Run 3A[3] DRE = 99.985%). Causes for this difference in MEK results are uncertain but may include contamination of the tubes with MEK and analysis performed by different labs.

The number of blanks taken and analyzed exceeded the goal of ten set in Table 7-1. Thirteen blanks were taken to determine if contamination had occurred and, if so, when the contamination occured during sampling and analyses. As shown by blanks values reported in Table A-2 of Appendix A, little or no contamination

TABLE 7-4. POHC SAMPLING AND ANALYSIS OF STACK GAS QA/QC RESULTS

Number of split samples	Split sample results[a] Run number	POHC	Result Wout, g/min x 10^{-3} 1	2	Average, g/min x 10^{-3}	Relative difference, %	No. of blank samples	No. of spiked samples	No. of standard samples	Completeness goals, % (No. of actual samples taken and analyzed/ goals x 100)
1 (within lab)	3D and 3D[1]	MEK	0.343	2.53	1.44	152	13[b]	43[c]	4 x 5[d]	81 (25/31 prs)
		MIBK	1.90	1.81	1.86	4.9				
		Perc	0.306	0.328	0.317	6.9				
		Toluene	12.3	12.0	12.2	2.5				
2 (between labs)	3A and 3A[3]	MEK	1.13	37.3	19.2	188				
		MIBK	3.88	2.75	3.32	34				
		Perc	0.508	0.393	0.451	25				
		Toluene	5.39	13.6	9.5	86				
3 (between labs)	3B and 3B[3]	MEK	2.35	80.4	41.4	188				
		MIBK	1.92	3.38	2.65	55				
		Perc	0.259	0.253	0.256	2.3				
		Toluene	17.1	12.3	14.7	33				

[a] Simultaneous side-by-side sample runs, 1 within MRC lab, 2 split with QA contractor.

[b] Includes two field pairs (one pair to MRC, one to QA contractor), four trip blanks, two lab and three baseline samples for total of 13 samples.

[c] Tube spiked with internal standard, 25 front tubes and 18 back tubes.

[d] Four POHC external standards run at five concentration levels.

occurred in transport or in the field tubes that would result in tube mass values above the background or baseline levels for any of the POHCs.

Each tube was spiked prior to analysis with 10 ng of benzene-d_6 internal standard. The internal standard was monitored during the analysis to check GC/MS performance. Forty-three samples were analyzed, excluding blanks; this included twenty-five front tubes and eighteen back tubes. Initially, nine back tubes were analyzed and found to be $\leq$10% the mass of the front tubes, showing that breakthrough of the front tubes did occur. It was decided that no additional back tubes would be analyzed. After review of the data, however, an additional nine back tubes were selected from runs having high mass values on the front tubes and analyzed to verify nonbreakthrough of the front tubes by POHCs.

Four external standards, one for each POHC, were analyzed by GC/MS to determine the correct ion ratios and compound retention times for each POHC. Standards were prepared at five concentration levels by spiking the standard solutions of the analytes (POHCs) in methanol onto blank tubes and then desorbing in the same manner as the samples. A five-point calibration curve was established for each POHC which correlated response/ion ratios with concentrations. Periodically new external standards were prepared and analyzed to verify that the GC/MS system maintained operation on the calibration curve.

7.3 WASTE FUEL QA/QC RESULTS

Daily composite samples were collected and returned to the MRC Dayton Laboratory for analysis of POHCs and other organics by capillary GC/MS. All of the samples were analyzed for the four POHCs. Two additional samples, from Days 1 and 4, were analyzed for the top twenty components. Samples were prescreened for major

components and approximate concentration so that external standards could be prepared at approximately 2X, 1X and 0.5X the sample concentrations for POHCs and other components. Samples were quantified using major ion areas of external standards with correction for internal standards where possible. If no external standards were available the total ion areas of external standards eluting nearest the retention time of the compound of interest were used to quantify the concentration.

Samples were diluted in either of two solvents, o-xylene (heavier solvent for the analysis of low boiling components) or methylene chloride (lighter solvent for the analysis of higher molecular weight compounds), and spiked with benzene-d_6 internal standard. Each sample was analyzed in duplicate for a total of four analyses per sample.

QA/QC results for waste fuel analyses are summarized in Table 7-5. All QA/QC goals for waste fuel sampling and analyses were met, including the number of splits and percent relative difference, the number of standards and blank taken and analyzed, and the completeness goals. Split results for both widescan and POHC analysis by GC/MS show excellant relative difference. Results for split samples on individual compounds for widescan and POHC are presented in Table A-3 of Appendix A. One waste fuel sample was split with the QA contractor, and results indicated relatively poor duplication between labs. QA contractor waste fuel results are shown in Table A-4 of Appendix A. The cause for the "between lab" discrepency is uncertain.

Standards are used by the GC/MS system for quantifying compound concentrations, and results are not calculated for percent recovery. Quantifying compounds and ions used are listed in Table A-3 of Appendix A.

TABLE 7-5. QA/QC RESULTS FOR WASTE FUEL ANALYSES

Number of samples (parameter)	Split sample results Relative difference, %			Standard sample results		Number of blank samples	Completeness goals, % (Number of actual samples/ goals x 100)	
	Range	Average	Standard deviation	Number of samples	Recovery, %			
42 (Wide-scan by GC/MS)	0.0-26.9	7.1	7.5	5	NA[a]	1[b]	100	(2/2)
30 (POHCs by GC/MS)	0.8-26.0	9.9	6.6	5	NA[a]	1[b]	100	(5/5)
1 (POHCs by GC/MS[c])	33.3-129.8	69.4	52.6	NA[d]	NA[d]	NA[d]	NA[d]	

[a]Not applicable; each constituent in the waste fuel is quantified by a separate external standard of the same chemical structure (listed in Table A-3). Percent recovery is not calculated.

[b]Solvent blank.

[c]Between lab split (MRC and EPA QA contractor).

[d]Not applicable.

7.4 CHLORINE, SULFUR, HEXAVALENT CHROMIUM, CHROME AND LEAD QA/QC RESULTS FOR ALL SAMPLES

Table 7-6 shows the QA/QC results for the analyses of chlorine, sulfur, Cr^{+6}, Cr, and Pb in various samples. In comparison with the test goals, most of the QA/QC results were met.

No QA/QC goals were set for chlorine and sulfur prior to the test.

No splits or spikes were analyzed for chromium and lead analyses; two each were scheduled. Two blanks were taken as specified. Three standards were analyzed and ranged ±36%.

Fewer blanks, splits, end spikes were analyzed for hexavalent chromium than scheduled. Results of splits and standards were within the limits specified prior to the test.

7.5 QA/QC RESULTS FOR TRACE METALS ANALYSES

All samples were prepared by wet-ashing with nitric acid, diluting to a known value, and then analyzing by ICP according to EPA Interium Method 200.7. The samples analyzed for trace metals included the waste fuel (solid and liquid phases), coal, clay feed, aggregate product, scrubber influent water, scrubber effluent water (liquid portions), and particulate matter (from EPA Method 5 filter and probe wash). A total of fifty samples were analyzed.

Forty-two reference standards were analyzed, two for each metal. Results of reference standards analysis are shown in Table 7-7. The average percent recovery is 99% ± 14.4% (95% confidence limits), which is within the limits set in Table 7-2; the range is 84% to 112%.

Three duplicate analysis were made, less than the eight splits scheduled in Table 7-1. Results are shown in Table 7-8. For all

TABLE 7-6. QA/QC RESULTS FOR CHLORINE, SULFUR, CHROMIUM, LEAD, AND HEXAVALENT CHROMIUM

	Split sample results				Standard or spiked sample results					
Parameter	Units	Result 1	Result 2	Relative difference, %	Units	Observed value	Actual value or (spiked value)	Sample value[a]	Recovery, %	Total samples analyzed[c]
Chlorine	%	1.05	1.10	4.7						30
	µg/g	<0.50	<0.50	0.0						
	µg/g	<0.50	<0.50	0.0						
	µg/g	<0.50	<0.50	0.0						
	mg/L	53.7	59.8	10.7	mg/L	241	(200)	101	70	
	mg/L	227	278	20.2	mg/L	598	(200)	368	115	
Sulfur	%	0.402	0.415	3.2						
	µg/g	3.64	3.10	16.0						30
Chromium[b]					ppm	0.011	0.014	-[a]	78.6	5
Lead[b]					ppm	0.500	0.390	-	128	12
					ppm	0.232	0.170	-	136	
Cr^{+6}	µg/L	298	298	0.0	µg/L	298	300	-	0.7	5
					µg/L	98	100	-	2.0	
					µg/L	198	200	-	1.0	

[a] For spiked sample only.

[b] By atomic absorption; samples separated into sludge and liquid portions for analyses.

[c] Excluding QA/QC and blanks.

TABLE 7-7. QA/QC RESULTS FOR TRACE METALS ANALYSES - REFERENCE STANDARDS

Metal	Sample 1			Sample 2		
	Observed value, µg/mL	True value, µg/mL	Recovery, %	Observed value, µg/mL	True value, µg/mL	Recovery %
Aluminum	1.1	1.0	106	0.98	1.0	96
Boron	1.1	1.0	112	0.43	0.43	100
Barium	0.88	1.0	88	1.2	1.0	84
Beryllium	0.98	1.0	98	0.35	0.35	100
Calcium	0.96	1.0	96	0.99	1.0	99
Cadmium	0.99	1.0	99	0.34	0.32	106
Cobalt	0.93	1.0	93	0.39	0.37	105
Chromium	1.0	1.0	102	0.32	0.30	107
Copper	0.92	1.0	92	0.72	0.70	103
Iron	1.1	1.0	110	0.54	0.53	102
Magnesium	0.89	1.0	89	1.1	1.0	105
Manganese	0.96	1.0	96	0.48	0.48	100
Molybdenum	1.1	1.2	94	0.51	0.50	102
Sodium	44	50	87	NA	NA	NA
Nickel	0.94	1.0	94	0.56	0.53	106
Lead	1.1	1.0	108	0.32	0.31	103
Tin	11	10	107	NA	NA	NA
Titanium	0.97	1.0	97	0.53	0.53	100
Vanadium	0.97	1.0	97	0.73	0.71	103
Yttrium	10	10	100	NA	NA	NA
Zinc	0.92	1.0	92	0.31	0.32	97

TABLE 7-8. QA/QC RESULTS FOR TRACE METALS ANALYSES - DUPLICATE SAMPLES

Metal	Coal No. 5, µg/gram			Aggregate No. 3			Waste fuel No. 4		
	1st value, µg/g	2nd value, µg/g	Rel. diff., %	1st value, µg/g	2nd value, µg/g	Rel. diff., %	1st value, µg/mL	2nd value, µg/mL	Rel. diff., %
Aluminum	4,460	4,470	0.2	12,000	12,100	0.8	ND	ND	0
Boron	24.1	23.4	3.0	4.42	3.93	10.8	5.47	5.47	0
Barium	62.1	61.7	0.6	23.5	23.5	0	ND	ND	0
Beryllium	0.725	0.706	3.0	0.595	0.619	4.0	ND	ND	0
Calcium	1,740	1,740	0.0	1,250	1,250	0	236	213	10
Cadmium	ND	ND	0	ND	ND	0	0.66	ND	0
Cobalt	4.7	3.52	29	6.81	5.77	16.5	5.32	4.76	11
Chromium	13.1	12.6	3.9	28.7	29.0	1.0	1.25	ND	0
Copper	16.8	15.3	9.4	13.8	10.7	25	16.5	19.1	15
Iron	7,960	7,900	0.8	8,790	8,880	1.0	5.10	4.74	14
Magnesium	452	451	0.2	1,360	1,380	1.5	10.6	9.29	13
Manganese	16.7	16.7	0	30.8	30.7	0.3	0.22	0.15	38
Molybdenum	ND	ND	0	ND	ND	0	ND	ND	0
Sodium	2,570	2,740	6.4	3,120	2,930	6.3	394	ND	0
Nickel	ND	ND	0	9.36	ND	0	ND	ND	0
Lead	ND	ND	0	15.8	14.5	8.6	10.9	9.62	13
Tin	ND	ND	0	ND	ND	0	ND	ND	0
Titanium	283	285	0.7	274	274	0	ND	ND	0
Vanadium	20.9	20.5	1.9	18.3	17.5	4.5	ND	ND	0
Yttrium	4.30	4.34	0.5	6.76	6.46	4.5	ND	ND	0
Zinc	5.32	5.07	4.8	22.2	22.5	1.3	15.1	16.1	6.4

three splits, the average relative difference was 4.2% ± 14.8% (95% confidence limits) for all metals, and the range was 0% to 37%; these values are within the limits specified in Table 7-1.

Three blanks were analyzed: one EPA 5 filter, one EPA 5 probe (acetone) wash, and one laboratory blank. This is less than the number of blanks specified in Table 7-1. In almost every case, the analyses of blanks showed that the trace metal was not detected by the ICP system. If a metal was detected, the concentration in the blank was always less than 0.035% of the sample concentration. Table 7-9 shows the results of the analyses of three blanks.

Table 7-9 also shows the limit of quantification (LOQ) for each metal. The LOQ is multiplied by the dilution factor to obtain the detection limit for a specific sample.

7.6 QA/QC RESULTS FOR EP TOXICITY ANALYSES

All samples followed the extraction procedure listed in the Federal Register. One duplicate, reference standard, and spike were analyzed for QA/QC with results shown in Table 7-10. QA/QC are good except for high recovery for barium reference standard and a high relative difference for the mercury duplicate.

TABLE 7-9. QA/QC RESULTS FOR TRACE METAL ANALYSES, BLANK ANALYSES AND LOQ's

Metal	Concentration			
	Blank filter, µg/g	Blank probe wash, µg/mL	Lab blank, µg/mL	LOQ, µg/mL
Aluminum	0.557	ND	ND	0.085
Boron	0.090	0.031	0.027	0.025
Barium	ND[a]	ND[a]	ND[a]	0.015
Beryllium	ND	ND	ND	0.002
Calcium	3.72	2.40	3.92	0.020
Cadmium	ND	ND	ND	0.012
Cobalt	ND	ND	ND	0.022
Chromium	ND	ND	ND	0.025
Copper	ND	ND	ND	0.030
Iron	0.148	0.088	0.129	0.017
Magnesium	0.342	0.170	0.241	0.010
Manganese	0.002	ND	0.003	0.003
Molybdenum	ND	ND	ND	0.080
Sodium	ND	ND	ND	5.0
Nickel	ND	ND	ND	0.065
Lead	ND	ND	ND	0.120
Tin	ND	ND	ND	0.450
Titanium	0.082	ND	ND	0.010
Vanadium	ND	ND	ND	0.030
Yttrium	ND	ND	ND	0.010
Zinc	0.010	0.027	0.043	0.010

[a]ND = not detected [or ≤ detection limit (LOQ)].

TABLE 7-10. QC RESULTS FOR EP TOXICITY ANALYSES

Compound	Value #1, µg/L	Value #2, µg/L	Relative difference, %	Result, µg/L	Actual value, µg/L	Recovery, %	Sample # Spiked	Spike Concentration, µg/L[b]	Recovery, %
Arsenic	62	84	30	33	55	60	4	400	84
Selenium	<50	<25	NA[c]	<25	26	NA	4	200	90
Mercury	8.1	1.8	126	2.4	1.9	126	4	1	77
Silver	<10	<10	NA	<10	0	NA	4	400	72
Barium	43,000	39,400	10	1,800	440	409	3	40,000	85
Cadmium	86	70	21	95	90	106	3	400	92
Chromium	30	50	50	265	250	106	5	100	130
Lead	4,833	6,333	27	110	115	96	3	1,000	98

[a] Sample #5.

[b] Sample spiked 50/50 ratio of sample/spike.

[c] NA = Not applicable.

Appendix A

VOST AND WASTE FUEL RAW DATA

Table A-1	VOST Sample Volumes
Table A-2	Blank, Front, and Back Mass Collected on VOST Tubes
Table A-3	Wide Scan and POHC Waste Fuel Data
Table A-4	QA Contractors Waste Fuel, and Scrubber Water Results

TABLE A-1. VOST SAMPLE VOLUMES

Run Number	Sample conditions[a] Volume (Vm), ft^3	Temperature (Tm), °F	Pressure (Pbar), in. Hg	Vm std dscf[b]	Vm std dscm[c]
1A	0.724	82	30.35	0.7126	0.02018
2A	0.723	64.5	30.15	0.7305	0.02069
2B	0.705	70.8	30.15	0.7038	0.01993
2C	Void				
2D	0.707	76.0	30.15	0.6990	0.01980
2E	Void				
2F	0.716	81.3	30.15	0.7010	0.01985
2G	0.706	84.0	30.15	0.6877	0.01948
2H	0.706	83.0	30.15	0.6890	0.01951
2I	0.709	83.0	30.15	0.6919	0.01960
2J	0.711	80.3	30.15	0.6974	0.01975
2K	0.708	80.0	30.15	0.6948	0.01968
3A	0.778	64.8	29.95	0.7804	0.02210
3A[3]	0.711	59.4	29.95	0.7370	0.02088
3B	0.846	72.5	29.95	0.8363	0.02369
3B[3]	0.834	64.0	29.95	0.8572	0.02427
3C	Field Blank				
3C[3]	Field Blank				
3D	0.682	72.0	29.95	0.6748	0.01911
3D[1]	0.705	64.0	29.95	0.7083	0.02006
4A	0.701	55.0	29.92	0.7158	0.02027
4B	0.700	59.8	29.92	0.7082	0.02006
4C	0.700	62.3	29.92	0.7048	0.01996
4D	0.696	64.0	29.92	0.6985	0.01978
4E	0.700	66.0	29.92	0.6999	0.01982
4F	0.700	70.0	29.92	0.6946	0.01967
4G	0.700	75.5	29.92	0.6874	0.01947
4H	0.700	79.0	29.92	0.6830	0.01934

(continued)

TABLE A-1 (continued)

Run Number	Sample conditions[a] Volume (Vm), ft^3	Temperature (Tm), °F	Pressure (Pbar), in. Hg	Vm std dscf[b]	Vm std dscm[c]
5A	0.700	59.5	29.90	0.7081	0.02006
5B	0.700	65.0	29.90	0.7007	0.01984
5C	0.700	66.0	29.90	0.6994	0.01981
5D	0.700	66.0	29.90	0.6994	0.01981
5E	0.700	69.0	29.90	0.6954	0.01969
5F	0.700	70.0	29.90	0.6941	0.01966

[a] [b] From VOST data sheets.

$$\text{Vm std dscf} = \text{Vm } Y \frac{Tstd}{Tm} \frac{Pbar}{Pstd}$$

where Y = 0.996 for all runs (except 3A[3], 3B[3], 3C[3] Y = 1.019)
Tstd = 528°R
Pstd = 29.92 in. Hg.

[c] 35.31 ft^3 = 1 m^3.

TABLE A-2. RESULTS OF THERMAL DESORPTIONS GC/MS ANALYSIS OF VOST TUBES FOR FOUR POHCs

Compound	L.O.D., ng[a]	Total nanograms on blank tubes									Average of all blanks
		Baseline blanks			Trip blanks		Field blanks		Laboratory blanks		
					2025	2094	3C-F	3C-B	3140	2163	
Methyl ethyl ketone	0.2	9.5	11.3	16.3	6.92	7.69	6.38	7.91	5.55	6.92	8.72
Methyl isobutyl ketone	0.3	ND[b]	ND	ND	ND	0.41	ND	1.15	4.93	ND	0.92[c]
Tetrachloroethylene (Perc)	0.2	NA[d]	NA	NA	2.1	0.58	0.69	5.52	1.82	0.97	1.23[c]
Toluene	0.1	0.69	0.82	0.86	3.91	4.06	2.59	35.3	2.74	3.83	2.44[c]

(continued)

TABLE A-2 (continued)

Compound	Total nanograms on front tubes														
	1A-F	2A-F	2B-F	2D-F	2F-F	2G-F	2H-F	2I-F	2J-F	2K-F	3A-F	3B-F	3C-F	3D-F	3D'-F
Methyl ethyl ketone	Plug[e]	Plug	Plug	Plug	23.2	17.7	25.6	5.62	8.54	12.1	83.6	144.5	6.38	24.7	132.5
Methyl isobutyl ketone					12.7	18.6	20.9	0.58	2.13	6.50	209.9	112.1	ND	89.7	89.6
Tetrachloroethylene (Perc)					ND	8.43	5.27	1.73	3.97	5.32	28.6	16.2	0.69	15.5	17.3
Toluene					0.67	114.7	171.1	2.95	94.1	84.4	293.1	318.9	2.59	577.2	588.0

	4A-F	4B-F	4C-F	4D-F	4E-F	4F-F	4G-F	4H-F	5A-F	5B-F	5C-F	5D-F	5E-F	5F-F
Methyl ethyl ketone	346.1	231.0	138.6	151.4	134.1	151.2	165.6	40.5	35.8	42.9	1.26	-[f]	168.2	64.0
Methyl isobutyl ketone	927.3	553.2	106.3	281.3	181.8	152.7	393.9	202.3	88.8	87.4	76.3		177.2	84.8
Tetrachloroethylene (Perc)	26.3	17.0	13.0	12.6	9.0	7.96	10.1	4.90	11.9	8.0	11.6		19.3	11.7
Toluene	2,262.2 (saturated)[g]	1,543.7	654.6	968.0	760.0	115.1	570.0	683.1	562.0	171.8	498.0		1,015.9	207.8

(continued)

TABLE A-2 (continued)

Compound	Total nanograms on back tubes								
	3A-B	3B-B	3C-B	3D-B	3D'-B	5A-B	5B-B	5C-B	5F-B
Methyl ethyl ketone	10.2	10.2	7.91	10.3	10.1	9.59	9.1	11.0	12.9
Methyl isobutyl ketone	0.73	0.96	1.15	2.75	1.29	0.81	ND	ND	2.14
Tetrachloroethylene (Perc)	1.03	0.98	5.52	1.38	1.41	1.65	1.46	1.78	1.61
Toluene	3.85	7.19	35.3	3.43	10.9	9.25	5.77	6.92	10.6

	Trip blank	Trip blank	4A-B	4B-B	4C-B	4D-B	4E-B	4F-B	4G-B	4H-B	5E-B
Methyl ethyl ketone	4.0	5.23	7.24	8.43	10.06	7.95	13.68	13.04	13.92	14.16	14.57
Methyl isobutyl ketone	ND	0.51	ND	ND	1.53	0.75	ND	ND	ND	ND	0.56
Tetrachloroethylene (Perc)	2.03	2.94	2.67	2.10	2.88	2.23	2.57	2.10	1.74	2.10	2.00
Toluene	3.23	5.20	40.12	16.53	22.39	7.20	8.54	7.96	7.02	10.04	6.73

(continued)

TABLE A-2 (continued)

Compound	Total nanograms on tubes						
	Lab						
	$3A^3$-F	$3A^3$-B	$3B^3$-F	$3B^3$-B	$3C^3$-F	$3C^3$-B	blanks
Methyl ethyl ketone	1,800	220	4,600	160	120	ND	ND
Methyl isobutyl ketone	140	ND	200	ND	ND	ND	ND
Tetrachloroethylene (Perc)	20	ND	15	ND	ND	ND	ND
Toluene	640	60	1,000	20	5	5	ND

[a]Limit of detection.

[b]Not detected; detection limit given by the L.O.D. for each compound.

[c]Omitting 4.93, 5.52, and 35.3 values.

[d]Not analyzed on baseline blanks because it was anticipated that trichloroethylene would be analyzed on tubes instead of tetrachloroethylene.

[e]Tube was plugged and could not be analyzed.

[f]Tube broke and could not be analyzed.

[g]Compound was present at such high concentration that the mass spectrometer was saturated. The value shown is lower than the actual concentration on the tube.

TABLE A-3. RESULTS OF CAPILLARY GC/MS ANALYSIS OF MAJOR COMPONENTS OF WASTE FUELS NUMBER 1 AND NUMBER 4 AND RELATIVE DIFFERENCE

Waste fuel component	Quantifying compound	Quantifying ion	Concentration, wt %: Number 1	Number 1 duplicate	Relative difference %	Concentration, wt %: Number 4	Number 4 duplicate	Relative difference %
Ethanol	Ethanol	(31)	1.62	1.49	8.4	1.77	1.88	6.0
2-Propanol	2-Propanol	(45)	4.88	4.21	14.7	1.94	2.00	3.0
1-Butanol	1-Butanol	(56)	1.73	1.82	5.1	0.666	0.873	26.9
Ethyl acetate	Ethyl acetate	(43)	0.703	0.632	10.6	0.697	0.749	7.2
Methyl ethyl ketone	Methyl ethyl ketone	(43)	2.02	2.05	1.5	2.52	3.09	20.7
Methyl isobutyl ketone	Methyl isobutyl ketone	(43)	1.54	1.51	2.0	1.09	1.15	5.4
Toluene	Toluene	(91)	8.28	8.52	2.9	7.85	8.26	5.1
Tetrachloroethylene	Tetrachloro-ethylene	(166)	0.190	0.200	5.1	0.068	0.072	5.7
Ethylbenzene	Ethylbenzene	(91)	1.21	1.26	4.0	2.20	2.37	7.4
Xylene (isomer No. 1)	o-Xylene	(91)	4.47	4.47	0.0	7.62	8.15	6.7
Styrene	Styrene	(104)	0.680	0.730	7.1	0.279	0.278	0.4
Xylene (isomer No. 2)	o-Xylene	(91)	1.30	1.29	0.8	2.42	2.62	7.9
2-Ethoxyethyl acetate	2-Ethoxyethyl acetate	(43)	2.03	2.03	0.0	1.14	1.26	10.0
C_3-Benzene[a] (isomer No. 1)	1,2,4-Trimethyl-benzene	(105)	0.470	0.470	0.0	0.325	0.342	5.1
C_3-Benzene[a] (isomer No. 2)	1,2,4-Trimethyl-benzene	(105)	0.570	0.570	0.0	0.330	0.379	13.8
C_{10}-Alkane[b] (isomer)	n-Decane	(57)	0.840	0.820	6.1	0.740	0.786	6.0
C_{11}-Alkane[c] (isomer)	n-Undecane	(57)	0.73	0.72	1.4	0.560	0.630	11.8
n-Propyl acetate	Methyl isobutyl ketone	(43)	1.51	1.49	1.3	0.988	1.03	4.2
2-Propanol, 1-(2-methoxy-1-methylethoxy)-isomer No. 1	n-Decane	Total ion	0.460	0.45	2.2	0.137	0.142	3.6
2-Propanol, 1-(2-methoxy-1-methylethoxy)-isomer No. 2	n-Decane	Total ion	0.50	0.68	4.1	0.147	0.176	17.9
2-Cyclohex 4-1-one, trimethyl (isomer)	n-Undecane	Total ion	1.33	1.23	3.8	0.514	0.569	10.2
Results for % R.D.								
No.					21			21
Sum					81.10%			185.0%
Mean, $\bar{x}$					3.86%			8.81%
Std. dev.					3.85%			6.39%
Range					0.0-14.7%			0.4-26.9%

[a] Compounds containing three carbons associated with a benzene ring.

[b] Compounds containing ten carbons associated with an alkane.

[c] Compounds containing eleven carbons associated with an alkane.

TABLE A-3 (continued)

Compound	Dilution solvent	L.O.D.[a], wt %	Solvent blank conc., wt %	Waste fuel No. 1 concentration, wt % Sample 1	Waste fuel No. 1 concentration, wt % Sample 2	Relative difference (Fuel No. 1) %	Waste fuel No. 2 concentration, wt % Sample 1	Waste fuel No. 2 concentration, wt % Sample 2	Relative difference (Fuel No. 2) %
Methyl ethyl ketone	Xylene	0.01	ND[b]	2.06	1.83	11.8	1.77	1.79	1.1
Methyl isobutyl ketone	CH_2Cl_2	0.01	ND	1.54	1.51	2.0	1.75	1.64	6.5
Tetrachloroethylene	Xylene	0.01	ND	0.191	0.147	26.0	0.143	0.170	22.9
Tetrachloroethylene	CH_2Cl_2	0.01	ND	0.190	0.200	5.1	0.243	0.230	5.5
Toluene	Xylene	0.01	ND	7.67	9.01	16.1	7.85	8.39	6.7
Toluene	CH_2Cl_2	0.01	ND	8.28	8.52	2.9	10.93	9.90	9.9

Compound	Waste fuel No. 3 concentration, wt % Sample 1	Waste fuel No. 3 concentration, wt % Sample 2	Relative difference (Fuel No. 3) %	Waste fuel No. 4 concentration, wt % Sample 1	Waste fuel No. 4 concentration, wt % Sample 2	Relative difference (Fuel No. 4) %	Waste fuel No. 5 concentration, wt % Sample 1	Waste fuel No. 5 concentration, wt % Sample 2	Relative difference (Fuel No. 5) %
Methyl ethyl ketone	1.88	1.78	5.5	2.52	3.09	9.2	3.97	4.53	13.2
Methyl isobutyl ketone	1.32	1.49	12.1	1.09	1.15	5.4	3.72	4.08	9.2
Tetrachloroethylene - Xylene	0.137	0.163	17.3	0.044	0.051	14.7	0.012	0.010	18.2
Tetrachloroethylene - CH_2Cl_2	0.178	0.212	17.4	0.068	0.072	5.7	0.048	0.052	8.0
Toluene - Xylene	7.99	7.89	1.3	7.43	8.41	12.4	7.75	7.69	0.8
Toluene - CH_2Cl_2	7.73	9.24	17.8	7.85	8.26	5.1	7.10	7.62	7.1

[a] ND - limit of detection.

[b] Not detected (or less than L.O.D.).

Totals this page for % R.D.

No. = 30

Sum = 296.90%

Mean, $\bar{x}$ = 9.90%

Std. Dev. = 6.59%

Range = 0.8-26.0%

TABLE A-4. CONTRACTOR WASTE FUEL AND SCRUBBER WATER ANALYTICAL RESULTS

Parameter	Units	Concentration
Waste fuel No. 3[a]		
Methyl ethyl ketone	wt %	1.3
Methyl isobutyl ketone	wt %	0.3
Toluene	wt %	13
Perc	wt %	NA[b]
Scrubber water influent No. 3		
Methyl ethyl ketone	ng	20
Methyl isobutyl ketone	ng	ND[c]
Toluene	ng	5
Perc	ng	NA
Blank	ng	ND[d]
Metals		
Cr^{+6}	ppm	<0.5
Cr (Total)	ppm	<0.5
Pb	ppm	<0.5
Scrubber water discharge No. 3		
Methyl ethyl ketone	ng	320
Methyl isobutyl ketone	ng	ND
Toluene	ng	420
Perc	ng	NA
Metals		
Cr^{+6}	ppm	<0.5
Cr (Total)	ppm	<0.5
Pb	ppm	16.0

[a]Only roughly quantified to assist in VOST analysis.

[b]NA - not analyzed.

[c]ND - not detected.

[d]ND - not detected for any POHC in blank.